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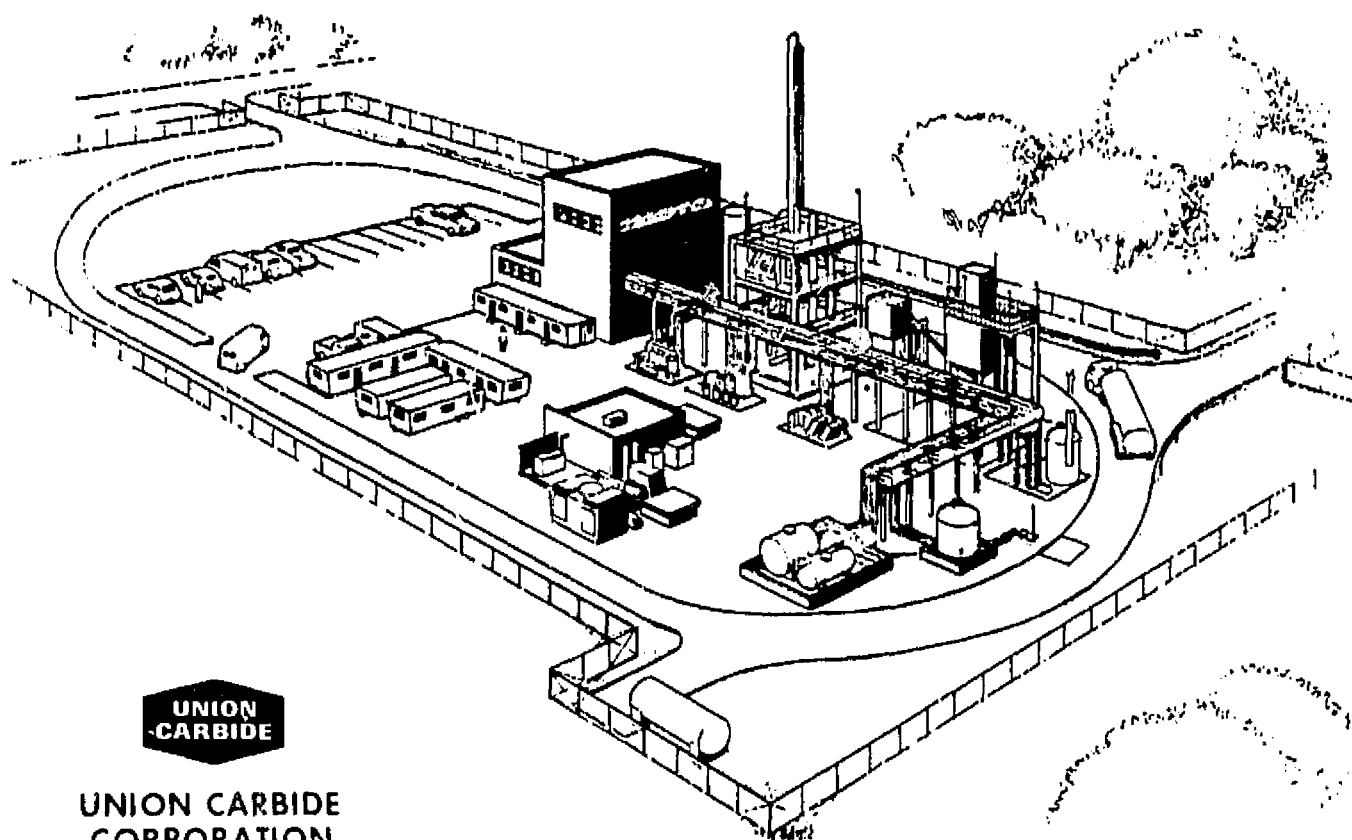
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QUARTERLY PROGRESS REPORT

JAN - MAR 1981

low cost solar array project

EXPERIMENTAL PROCESS SYSTEM DEVELOPMENT UNIT FOR
PRODUCING SEMICONDUCTOR-GRADE SILICON USING THE
SILANE-TO SILICON PROCESS



UNION CARBIDE
CORPORATION

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ABSTRACT

This report covers work performed in January, February, and March, 1981 on JPL/DOE Contract 954334, Phase III. This phase consists of the engineering design, fabrication, assembly, operation, economic analysis, and process support R&D for an Experimental Process System Development Unit (EPSDU).

The civil construction work was completed and the mechanical bid package is in preparation and will be issued during the next quarter. The electrical design effort is in progress.

Parallel efforts which complement the mechanical design are the process flow diagrams and control instrumentation logic for startup operation and shutdown. These are in progress and will identify all process and utility streams, control systems, and flow logic.

An additional key effort which is progressing well is the data collection system development. This system takes the signals from the instrumentation, translates them into engineering units and finally develops a data report which summarizes all key performance parameters. Direct readout of selected data can be observed at any time. Storage and retrieval of 330 compositional analyses are taken of 63 process streams for off-line analyses.

Equipment has been arriving at the EPSDU site daily and a comprehensive "material receiving log" is being maintained. In addition, an equipment tagging procedure has been set up to account for all items received on site.

Cleaning procedures have been established to assure a contamination-free product and inspection visits have been made to the fabricators of specialty equipment.

The free-space reactor experimental work completed in the last quarter has been reported and the results are summarized in this report. Overall, the objectives were achieved and the unit can be confidently scaled to the EPSDU size based on the experimental work and supporting theoretical analyses.

The fluidized-bed reactor activity has proceeded to a point where startup and checkout can be started, piping and instrumentation was completed in this quarter.

Progress of the shotter/melter system at Kayex was slowed due to numerous difficulties in obtaining components made to the desired specifications. Initial checkout runs have been made but further modifications are required to be able to deliver shot.

In summary, work is progressing well and the EPSDU facility is showing signs of accomplishments as equipment is received.

SECTION I. INTRODUCTION

This report covers work performed in January, February and March, 1981 on JPL/DOE Contract 954334, Phase III.

The overall objective of the LSA Silicon Material Task is to establish a chemical process for producing silicon at a rate and cost commensurate with the production goals of the LSA project for solar-cell modules. This material must be suitable for utilization in the large-area sheet process and in the automated process for the fabrication of solar cells having satisfactory physical and electrical performance characteristics.

As part of the overall Silicon Material Task, Union Carbide developed the silane-silicon process and advanced the technology to the point where it has a definite potential for providing high-purity polysilicon on a commercial scale at a price of \$14/kg by 1986 (1980 dollars). This work, completed under Phases I and II of the contract, provided a firm base for the Phase III Program (initiated in April 1979) aimed at establishing the practicality of the process by pursuing the following specific objectives:

- Design, fabricate, install, and operate an Experimental Process System Development Unit (EPSDU) sized for 100 MT/Yr to obtain extensive performance data to establish the data base for the design of commercial facilities.
- Perform supporting research and development to provide an information base usable for the EPSDU and for technological design and economic analysis for potential scale-up of the process.
- Perform iterative economic analyses of the estimated product cost for the production of semiconductor-grade silicon in a facility capable of producing 1000 MT/Yr.

This process for preparing semiconductor-grade silicon in the EPSDU from metallurgical-grade (M-G) silicon is based on a well-integrated arrangement of purification steps that provides a cost-effective process system.

The three basic steps entail converting M-G silicon to trichlorosilane, redistributing the trichlorosilane to produce silane, and thermally decomposing the silane to form silicon powder. The powder is then melted and the molten silicon is cast into polycrystalline silicon for subsequent use in fabricating solar cells.

The technical progress presented in this report is arranged according to the Work Breakdown Structure (WBS) shown in Table I.

1.1 DESIGN/PROCUREMENT

1.1.1 PROCESS DESIGN

- 1.1.1.1 SILAGE PROCESS DESIGN
- 1.1.1.2 ENG. DESIGN SUPPORT (SILAGE)
- 1.1.1.3 RUNNER TESTS
- 1.1.1.4 PYROLYSIS/MELTING PROCESS DESIGN
- 1.1.1.5 ENG. DES. SUPP. (PYROLYSIS/MELTING)

1.1.2 FACILITY DESIGN

- 1.1.2.1 LAYOUT, SITE EVALUATION
- 1.1.2.2 ENVIRONMENTAL & SAFETY
- 1.1.2.3 GANTRY SCALE MODEL
- 1.1.2.4 OPERATIONS LIAISON
- 1.1.2.5 FACILITY SCALE MODEL
- 1.1.2.6 GANTRY SCALE-MODEL UPDATE

1.1.3 EQUIP. DESIGN, SPECS, PROCUREMENT

- 1.1.3.1 PROCESS CONTROL
- 1.1.3.2 ELECTRICAL POWER SYSTEM
- 1.1.3.3 PROCESS EQUIP. UCC DESIGNED
- 1.1.3.4 PROCESS EQUIP. VENDOR DESIGNED
- 1.1.3.5 AUXILIARY EQUIPMENT
- 1.1.3.6 SUPPORT EQUIPMENT
- 1.1.3.7 EQUIPMENT SPECIALTIES
- 1.1.3.8 DATA COLLECTION SYSTEM

1.1.4 INSTALL. DES., SPECS, SUBCONTRACT

- 1.1.4.1 SITE DRAWINGS & SPECS
- 1.1.4.2 CIVIL DESIGN, SPECS & MATERIALS
- 1.1.4.3 CIVIL DRAWINGS
- 1.1.4.4 MECH. DESIGN, SPECS, & MATERIALS
- 1.1.4.5 MECHANICAL DRAWINGS
- 1.1.4.6 ELECT. DESIGN, SPECS, & MATERIALS
- 1.1.4.7 ELECTRICAL DRAWINGS

1.1.5 COST ESTIMATING

- 1.1.5.1 FACILITY COST ESTIMATE
- 1.1.5.2 ENGINEERING DESIGN SUPPORT

1.1.6 PYROLYSIS/MELTING SYSTEM DESIGN

- 1.1.6.1 CONTROL SYSTEM DESIGN
- 1.1.6.2 ELECTRICAL EQUIPMENT
- 1.1.6.3 FREE-SPACE REACTOR EQUIPMENT
- 1.1.6.4 OTHER EQUIPMENT
- 1.1.6.5 STRUCTURAL INSTALLATION DESIGN
- 1.1.6.6 MECHANICAL INSTALLATION DESIGN
- 1.1.6.7 ELECTRICAL INSTALLATION DESIGN

1.2 EQUIPMENT FABRICATION/DELIVERY

1.2.1 PROCESS CONT. & DATA-COLLECTION SYS.

- 1.2.1.1 FIELD INSTRUMENTS & CONTROLS
- 1.2.1.2 PANELS & PANEL INSTRUMENTS
- 1.2.1.3 DATA COLLECTION SYSTEM
- 1.2.1.4 OTHER PROCESS CONTROL EQUIPMENT

1.2.2 ELECTRICAL POWER SYSTEM

- 1.2.2.1 POWER SUPPLY
- 1.2.2.2 PUMP/CONTROL CENTER
- 1.2.2.3 OTHER POWER SYSTEM EQUIPMENT

1.2.3 PROCESS EQUIPMENT-LINE DESIGNED

- 1.2.3.1 Unused No.
- 1.2.3.2 REACTORS
- 1.2.3.3 DISTILLATION COLUMNS
- 1.2.3.4 Unused No.

1.2.4 PROCESS EQUIPMENT-VENDOR DESIGNED

- 1.2.4.1 COMPRESSORS
- 1.2.4.2 PUMPS
- 1.2.4.3 HEAT EXCHANGERS
- 1.2.4.4 TANKS
- 1.2.4.5 SOLIDS HANDLING
- 1.2.4.6 WASTE TREATMENT
- 1.2.4.7 OTHER PROCESS EQUIPMENT

1.2.5 AUXILIARY EQUIPMENT

- 1.2.5.1 THERMAL HEATER
- 1.2.5.2 REFRIGERATION UNIT
- 1.2.5.3 COOLING TOWER
- 1.2.5.4 INSTRUMENT AIR UNIT
- 1.2.5.5 Unused No.
- 1.2.5.6 OTHER AUXILIARY EQUIPMENT

1.2.6 SUPPORT EQUIPMENT

- 1.2.6.1 O/C TRAILER
- 1.2.6.2 Unused No.
- 1.2.6.3 Unused No.
- 1.2.6.4 Unused No.

1.2.7 PYROLYSIS SYSTEM

- 1.2.7.1 PYROLYSIS EQUIPMENT
- 1.2.7.2 OTHER PYROLYSIS/MELTER SYS. EQUIP.

1.2.8 PROCUREMENT SYSTEM

- 1.2.8.1 PURCHASING, EXPEDITING
- 1.2.8.2 EQUIPMENT DESIGN, INSPECTION
- 1.2.8.3 VENDOR PRINT CONTROL
- 1.2.8.4 EQUIPMENT CLEANING

1.3 INSTALLATION/CHECKOUT

1.3.1 INSTALLATION SUB-CONTRACTS

- 1.3.1.1 SITE
- 1.3.1.2 CIVIL, UNDERGROUND, STRUCTURAL
- 1.3.1.3 Unused No.
- 1.3.1.4 MECHANICAL & PIPING
- 1.3.1.5 ELECTRICAL
- 1.3.1.6 Unused No.
- 1.3.1.7 OTHER SUBCONTRACTS
- 1.3.1.8 PYROLYSIS/MELTING SYSTEMS

1.3.2 INSTALLATION MATERIALS

- 1.3.2.1 Unused No.
- 1.3.2.2 MECHANICAL, PIPING
- 1.3.2.3 Unused No.
- 1.3.2.4 OTHER INSTALLATION MATERIALS

1.3.3 INSTALLATION SUPPORT

- 1.3.3.1 FIELD SUPERVISION
- 1.3.3.2 TECHNICAL SUPPORT
- 1.3.3.3 Unused No.
- 1.3.3.4 Unused No.
- 1.3.3.5 PROCUREMENT

1.3.4 CHECKOUT

- 1.3.4.1 PROCESS & EQUIPMENT
- 1.3.4.2 SAFETY, ENVIRONMENTAL
- 1.3.4.3 PROCESS CONTROL, DATA COLL. SYS.
- 1.3.4.4 Unused No.
- 1.3.4.5 Unused No.

1.4 OPERATION

1.4.1 PREPARATION

- 1.4.1.1 OPERATING MANUAL
- 1.4.1.2 TRAINING
- 1.4.1.3 TEST PLAN

1.4.2 START-UP

- 1.4.2.1 SYSTEM DRYING
- 1.4.2.2 WASTE TREATMENT
- 1.4.2.3 HYDROGENATION
- 1.4.2.4 DISTILLATION
- 1.4.2.5 PYROLYSIS/COMB.

1.4.3 MODIFICATION

- 1.4.3.1 DESIGN
- 1.4.3.2 PROCUREMENT
- 1.4.3.3 INSTALLATION

1.4.4 INITIAL FEASIBILITY

- 1.4.4.1 PREPARATION
- 1.4.4.2 STEADY-STATE
- 1.4.4.3 SAMPLE PREPARATION

1.4.5 DATA GENERATION & ANALYSIS

- 1.4.5.1 HYDROGENATION
- 1.4.5.2 DISTILLATION/RECRYSTALLIZATION
- 1.4.5.3 WASTE TREATMENT
- 1.4.5.4 POLYCRYSTALLINE

1.4.6 PYROLYSIS & CONSOLIDATION

- 1.4.6.1 FREE-SPACE REACTOR
- 1.4.6.2 MELTER/SHOTTER

1.4.7 OFF-SITE QC ANALYSIS

- 1.4.7.1 SAMPLE PREPARATION
- 1.4.7.2 ANALYSIS

1.4.8 FINAL PERFORMANCE EVALUATION

- 1.4.8.1 PREPARATION
- 1.4.8.2 OPTIMIZED PERIOD
- 1.4.8.3 SAMPLE DELIVERY

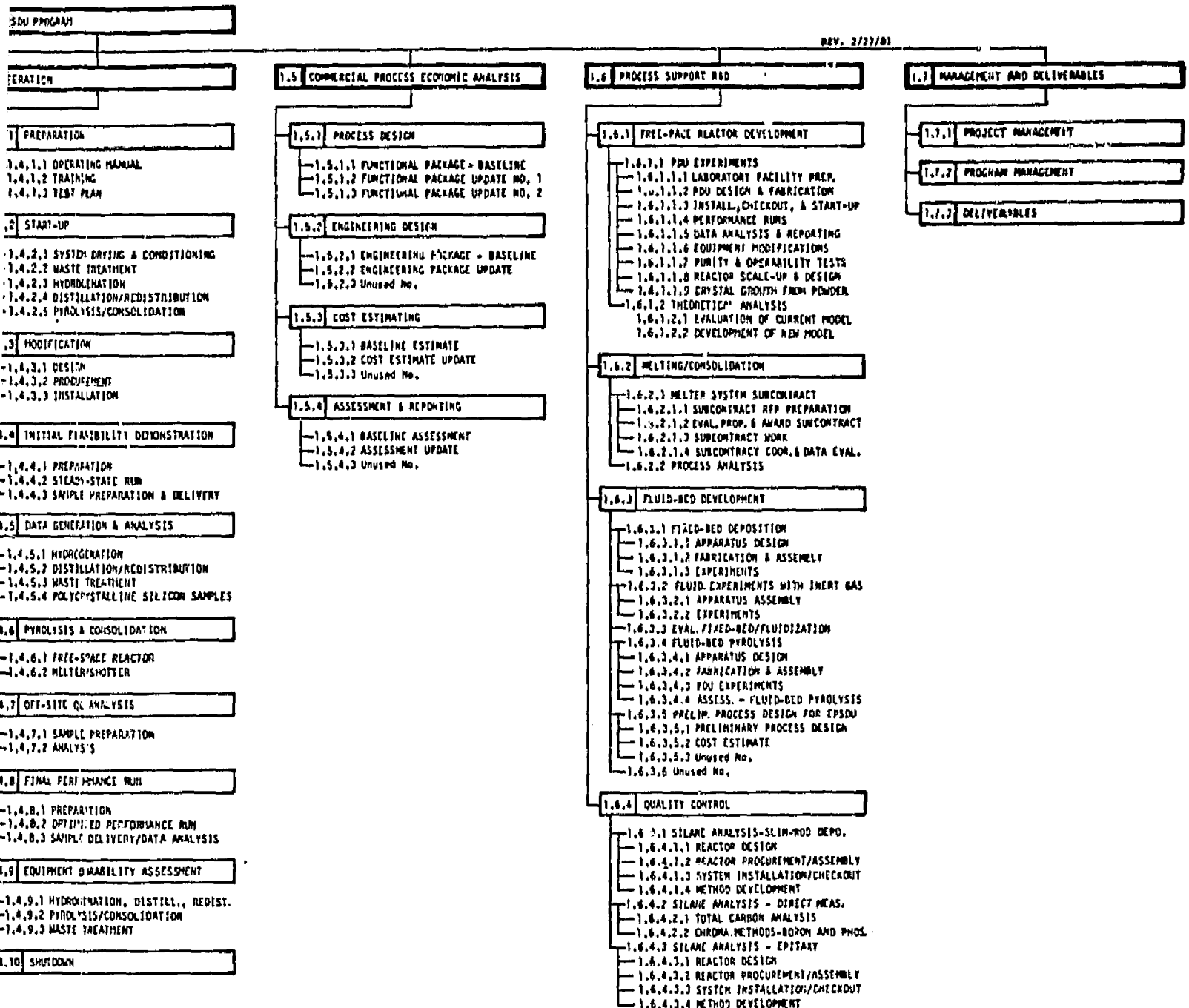
1.4.9 EQUIPMENT DURABILITY

- 1.4.9.1 HYDROGENATION
- 1.4.9.2 PYROLYSIS/COMB.
- 1.4.9.3 WASTE TREATMENT

1.4.10 SHUTDOWN

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TABLE I
WORK BREAKDOWN STRUCTURE
REVISED FOR FY81, 82, & 83



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SECTION II. TECHNICAL ACTIVITIES (BY WBS NUMBER)

1. EPSDU PROGRAM

As illustrated in Table I, the current Phase III Program consists of seven primary (WBS level 2) divisions of effort:

- EPSDU Design and Procurement
- EPSDU Equipment Fabrication and Delivery
- EPSDU Installation and Checkout
- EPSDU Operation
- Commercial (1000 MT/Yr) Process Economic Analysis
- Process R&D to Support EPSDU Design and Commercial Analysis
- Program Management

Collectively, these activities encompass all efforts required to attain the program objectives. The subdivisions (WBS levels 3, 4, and 5) define the individual work items that must be performed. The progress for this quarter, documented in this section, is reported at the work-item level. Only work items that are currently completed are included.

1.1 EPSDU DESIGN/PROCUREMENT

This effort includes all engineering, design, and procurement activities necessary to transform the process design, developed during the Phase II Program, into a complete installation-drawing package for EPSDU. The major tasks include process design updates, facility design, equipment design and procurement, installation design, and cost estimating support.

1.1.1 Process Design

The process design effort is geared toward using the most recent information available to provide the most practicable integration of process subsystems for attaining the EPSDU Program objectives. The process design package consists of a heat/mass balance, process description, process flow

diagram, and functional specifications for process equipment. The original package, issued in June 1979, served as the basis for the subsequent engineering effort. Beneficial data from the supporting R&D effort and other process-related analyses and experiments were used to update the original package. Process engineers, using information available from the Phase I and Phase II Programs, provide direct support to the facility and equipment design efforts.

1.1.1.2 Engineering Design Support

The key activities during this quarter were to provide support to the Engineering Department for piping requirements (definition, material, insulation), valve selection, cleaning procedures, instrumentation and control definition and logic of operation for the process including shutdown/startup procedures. The piping, valves, and instruments were arranged to permit the process equipment to function properly. Each valve in the system was reviewed and a cost effective selection was made. Specifications for safety relief valves and check valves were written, and purchase orders written. Assistance was provided for the Instruments and Controls Group by reviewing the logic to be employed during startup and shutdown of the various procedures.

The relay logic is basically done by process area: hydrogenation, distillation/redistribution, pyrolysis/consolidation, and waste treatment. The first two areas have the most complex relay logic. Automatic shutdowns and relay interlocks are used whenever rapid, programmable action is necessary to avoid potential injury to personnel or significant equipment damage.

A potential problem on insulation of the high temperature lines from the superheater to the hydrogenation reactor was reviewed and resolved by specifying a preformed insulation with a lower thermal conductivity and greater thickness than shown on the drawing. If the temperature drop is found excessive during checkout and startup, more insulation will be added to flanges and valves.

A consolidated cleaning procedure for inclusion into the design package was prepared for the cleaning of components used in chlorosilane service.

1.1.1.4 Pyrolysis/Consolidation Process Design

A new WBS Work Item was created for recently identified work associated with the free-space reactor R&D at Tonawanda and the silicon melting/shotting work at Kayex. Before these two technologies can be implemented at EPSDU, a silicon powder transport system and a product silicon (shot) removal/packaging system must be developed. Under normal conditions, this activity should begin after completion of these R&D programs.

As reported in the previous quarterly report, activity was delayed due to manpower requirements elsewhere. However, work was re-started in January and includes process design work not covered under existing R&D tasks. This effort is required for integration of silane pyrolysis and consolidation equipment into EPSDU.

A preliminary Process Flow Diagram was prepared which identifies all process and utility streams to and from various system components and includes mass balances and equipment list.

Several vendors had been contacted regarding pneumatic conveying of silicon powder produced in the free-space reactor. No commercially available system was identified which matched our needs. Reasons cited were small powder particle size, lack of knowledge with the powder flow properties, the use of hydrogen conveying gas, and the purity requirements of the process. However, the feedback from vendors was that dense-phase conveying was still attractive and that with some in-house testing of dense-phase conveying, the concept is feasible.

An evaluation of the powder transport test data, using the Wen and Simons model predicted that a 1-1/2 inch diameter conveying line is required. An internally polished stainless steel sanitary tube appears to be the best compromise between conveying gas requirement, conveying pressure drop, sensitivity to plugging, and mass throughput. A one-half ton-per-hour rate seems to be the most likely place to operate, although a rate of almost twice that is possible.

The current mechanical drawings for the EPSDU free-space pyrolysis reactor and Kayex-designed shotting equipment were reviewed. Recommendations were made for improvement on the free-space hopper design, layout, the shotter purge gas flow control, and applicability of the argon purged feed hopper in EPSDU. Some retrofitting of the auger feed hopper will have to be made, in order to accommodate a production throughput of powder and to match up with the free-space reactor.

A partial preliminary process design package was delivered to the Engineering Department and consists of a process flow diagram, process description, heat and mass balance, equipment functional specifications and preliminary equipment layout. Final equipment layout drawings will be prepared during the second quarter. The EPSDU consolidation Building Equipment List is shown in Figure 1.

1.1.2 Facility Design

Facility design consists primarily of the effort required to translate the process design functional requirements into specific plans regarding site, physical arrangement, human factors, and safety and environmental considerations. Personnel who will operate the facility participate to provide human factor inputs and to become familiar with the process.

1.1.2.2 Environmental and Safety

The environmental effort includes the assessment for regulatory compliance of all environmental considerations associated with the process, preparation of standards, and obtaining appropriate approvals and permits. The safety aspects include evaluation of the process and detailed design and monitoring of installation and operation to ensure that all features necessary for public and staff safety are included and proper procedures are used.

Safety review meetings were held to discuss remaining action items from the November safety team comments. Meeting minutes were issued showing most items implemented. A review of gas phase versus liquid phase silane storage was also held. Meeting minutes were issued which note that we continue to prefer and will utilize liquid phase storage at EPSDU.

The air discharge permit application has finally been formally approved as a permit-to-construct by the Indiana Air Quality Office for East Chicago.

EQUIPMENTDESCRIPTION

349-02	Valve Cleaning Enclosure
349-04	Pyrolysis/Consolidation Control Enclosure
441-08	Shot Receiver
441-10	Shotter Feed Hopper
441-18	Reactor Liner Cleaning Station
441-24	Hydrogen Accumulator
443-04	Shot Receiver Hydrogen Recycle Blower
444-04	Pyrolysis Hydrogen Precooler
444-06	Shot Receiver Hydrogen Recycle Cooler
445-02	Free-Space Reactor
446-02	Vacuum Pump
447-02	Vacuum Pump Suction Filter
447-04	Shot Receiver Hydrogen Recycle Filter
448-08	Powder Scale (P.O. #825-60067)
448-10	Shot Scale (P.O. #825-50068)
448-12	Shot Lockhopper
449-04	Shotter (P.O. #825-50079)
449-08	Acid Wash Exhauster
449-10	Drumming Station Hood
449-12	Free-Space Reactor Pyrolysis Furnace
449-16	Deionizer
449-18	Vacuum Dust Collector
449-20	FSR Hoists
649-02	Free-Space Transformer

ITEMS TO BE SPECIFIED BUT NOT PURCHASED AS CAPITAL EQUIPMENT

Shot Storage Drums
Excess Powder Drums
FSR Quartz Liners
Shotter Crucibles

Figure 1 EPSDU Consolidation Building Equipment List

1.1.3 Equipment Design, Specification, Procurement

The equipment related effort includes development of the control system, preparation of the piping and instrumentation diagram, preparation of wiring schematics and control panel drawings, and the design of equipment. The specification activity includes definition of specific requirements for each item of equipment, preparation of bid packages, evaluation of vendor quotation, and preparation of final specifications and drawings. Procurement includes the issuance of procurement packages to selected vendors and obtaining comprehensive design information necessary for preparing installation drawings.

The design and procurement of each item of equipment is accomplished through the combined efforts of process engineers, equipment engineers, and purchasing agents. These efforts produce a series of documents that evolve, ultimately, into a complete, definitive procurement package. The individual documents and their respective uses are as follows:

- Functional Specifications: This specification is developed by the process engineer based on process requirements reflected in the process flow diagram, heat/mass balance, and process control scheme. It defines the duty that this item of equipment must perform for the overall process system to operate.
- Engineering Specifications: Using the Functional Specification as a basis, the equipment engineer determines the specific type of equipment necessary to satisfy the process requirements. This translation of process requirements into hardware-specific information is delineated in the engineering specification.
- Request for Quotation (RFQ): The Request for Quotation (Form PUR 201), prepared by the equipment engineer, summarizes the equipment requirements, identifies vendors to be contacted, and defines the bidding instructions. This form, plus the engineering specification, constitutes the RFQ package submitted to the purchasing department for transmittal to potential vendors. Vendor quotations are reviewed by the equipment engineer and, based on a technical, cost and schedual evaluation, a vendor is selected.

- Request for Requisition (RFR): The Request for Requisition, prepared by the equipment engineer, consists of the RFQ package plus the Bid Evaluation Report that identifies the selected vendor and the specific equipment model to be purchased. The RFR is submitted to purchasing and serves as the technical basis for the purchase order.
- Purchase Order (PO): The Purchase Order (Form L334-31DO), prepared by the purchasing agent, definitizes the terms and conditions, delivery requirements, and billing instructions applicable to the particular equipment and vendor. This form supplements the technical information contained in the RFR to provide a complete procurement package. When the internal review cycle is completed and the appropriate approvals have been obtained, the validated Purchase Order is issued to the vendor.
- Procurement Status Report (PSR): After the order is placed technical performance is monitored by engineering personnel and contractual performance is monitored by purchasing personnel. Status is reflected in the Procurement Status Report. (The PSR for March, presented in Appendix A, reflects the current procurement status of all EPSDU equipment).

These six documents serve as milestones for measuring performance of the procurement cycle for each item of equipment.

1.1.3.1 Process Control

The controls systems engineering effort includes all activities associated with developing the P&I diagram, designing process control loops and control panels, specifying valves and instrumentation, and preparing control wiring and pneumatic tubing diagrams.

The Revision "G" version of the P&I diagram was issued. It is being used to finalize and check all equipment and piping drawings in the mechanical installation bid package. This is the last version of the P&I diagram to be issued prior to the actual start of equipment and piping installation work at EPSDU.

The process control design work which centers around the control logic diagram is in progress and will be finalized in the second quarter.

Control panels (6) and associated instrumentation were defined, quoted, and purchase orders issued.

1.1.3.3 Process Equipment, UCC Designed

A test was run on the silane distillation column packing to insure that the SCS-1 cleaning procedure would not alter the packing or result in any unusual gas evolution due to its high surface area. The results showed that the standard SCS-1 cleaning procedure as specified was acceptable.

1.1.3.4 Process Equipment-Vendor Designed

This activity includes the engineering effort associated with specifying and selecting process equipment such as compressors, pumps, and tanks that will be designed and fabricated by commercial suppliers.

Drawings were received and approved for all waste burner components except the flame supervision panel.

The refrigerant brine heater and the waste agglomerator were placed on order.

1.1.3.7 Other Equipment, Specialties

Key items addressed during this quarter were:

a. Mechanical Cleaner for the Agglomerator

A spring-type cleaner was chosen for the vertical tubes. These loosely wound springs will be friction-hung from the top feed-thru fitting of each agglomerator tube. Silica deposits will be scraped off by a vertical motion of the center which will be hand-stroked. The construction material is high-tensile steel piano wire.

b. Ram Valve Seal Pressurization Assembly

The ram valve seal pressurization assembly for the hydrogenation reactor was designed, sketched and components ordered.

c. Sample Conditioner

Fabrication of the chlorosilane sample conditioner has begun.

d. Stack Gas Analyzer

A commercial instrument was not available. It was decided that a discontinued Beckman unit can be tailored to our exact needs. The analyzer, contained in a metal box, draws a sample of the stack gas through a magnetically stirred, constant temperature bath containing de-ionized water. A chloride ion probe senses any hydrogen chloride present in the stack gas, sending a signal to the pH meter which has a meter relay alarm feature.

1.1.3.8 Data Collection System

This work item covers all effort to tailor and specify a computerized data-collection system for EPSDU.

The software preparation plan consists of the following steps:

- a. Prepare a flowsheet to diagram the algorithm, or logic path, for each routine or task.
- b. Review the necessary mini-computer library routines, or pre-programmed implementation programs, with our Computer Applications group to pass the data from the P.V. tables to the program being written. This may either be a simple addressing sequence or a more complex overlay of several library routines.
- c. Write the program in Data General Fortran, a modified Fortran IV, for specific use with the Data General compiler.

- c. Debug individual routines by inputting some values and checking the output with a calculator.
- d. Check the integrated system as it is structured for EPSDU to assure that the signals from the instrumentation are being correctly converted for data storage.

The content of the proposed EPSDU data report which summarizes the key performance parameters each day is as follows:

1. Specified primary process variables (approx. 50) vs clock time; one hour averages, daily averages and deviations
 - Flows on ACFM or GPM
 - Temperatures in °F
 - Pressures in psia
 - Levels in inches
2. Heat and Mass Balance
 - a. Overall facility Si/H/Cl balance
 - b. Sub-system balances
 - Hydrogenation reactor/superheater
 - Quench condenser loop
 - Hydrogenation sub-system
 - Stripper Column
 - TCS Column/TCS Redistribution Reactor
 - DCS Column
 - Silane Column/DCS Redistribution Reactor
 - Distillation sub-system
 - Pyrolysis/Consolidation sub-system
 - c. Minimum inclusion into daily report will be based on daily averages, but hourly averages can also be used as required.
3. Utility consumptions, absolute and pro-rata
 - Electrical power
 - Natural gas
 - Water
 - Nitrogen
 - Argon

4. Off-line/waste treatment inputs
 - a. Q. C. trailer off-line analyses
 - b. Process off-line inputs
 - c. Waste treatment area flows, differential pressures, and temperatures

Estimated length of daily report is 5 pages of process variables vs time plus 5 pages of other operations performance data.

The overall data collection scheme for EPSDU is to log data onto a series of disk packs for statistical reduction at Tonawanda on a large IBM computer, while on a daily basis examining the data taken at E. Chicago. This pre-screening of data at E. Chicago is important to highlight any questionable points, check instrumentation variation and guide the overall data collection program.

The following activities were in progress during the first quarter:

- Develop subroutine to fit properties of the system fluids to simple polynomial expressions
- Develop heat exchanger duty flowsheet
- Write main program to determine system heat and mass balance based on subprograms
- Calibrate and convert signals measured by thermocouples, flowmeters, etc., to a meaningful form for input to the main computer program
- Storage and retrieval of the 330 compositional analyses are taken on 63 process streams. The best method of storage/retrieval was worked out so that maximum usage is made of existing mini-computer software. These compositions must be handled differently from other process data because the analyses are not performed in real time, rather after a delay and also because each analysis is not required frequently.

- The design and programming of an archive system is 50% complete. This system allows process data to be sampled on any specified time frequency, in specified groups, for any specified time period. Up to 255 different groups can be defined with each group containing up to 250 variables. The system will maintain the data on-line for a period of 2 weeks, and off-line indefinitely.

Work which remains to be completed for the applications programming package is:

- (a) Incorporate the status variables in the mass balance for batch items. (Hydrogen lost each time the lock hopper is cycled or mass lost when the waste chlorine tank is cycled).
- (b) Detailing the conversational off-line entry from the Q.C. trailer.
- (c) Definition and documentation of the archive system by the in-house Computer Applications Group.
- (d) Integrate/Interface with the archive system.

1.1.4 Installation Design, Specification, Subcontract

This design effort includes development of separate installation drawing packages for the site, civil, mechanical, and electrical specialties based on the engineering design effort and vendor-supplied information. Specification activity includes definition of specific requirements for performing all installation functions. Subcontracting includes the preparation of bid packages, evaluation of quotes, sub-contractor selection and contract negotiation.

1.1.4.4 Mechanical Design, Specification & Materials

An index of piping and insulation specifications for process and utility lines was completed.

Work continues on the field bill of materials which identifies each component in the EPSDU facility and to determine whether Linde or the mechanical installation subcontractor is responsible for its supply. This is an imposing document which will number several hundred pages when completed in mid-April.

Procedural specifications for pressure testing and leak testing chlorosilane and silane piping were completed.

Procedural specifications for cleaning fabricated assemblies of silane piping were developed, reviewed and finalized. A similar, but less stringent specification for chlorosilane piping was developed and reviewed, and will be finalized in early April.

Requisitioning piping components and materials should be completed by mid-April. These include valves, gaskets, filters, pressure and temperature indicators etc.

Forty-four separate vendor RFR's have been issued, a substantially greater number of requisitions than originally planned. Rather than assign responsibility to the mechanical contractor for purchasing many typically standard piping components and materials, we have elected to purchase these items to ensure proper type, expedite delivery, or because the components require special chlorosilane/silane cleaning at Tonawanda.

An engineering team meeting has been scheduled for April 14, 1981 to review the Mechanical Bid Package prior to its scheduled release for bids on April 24, 1981. This review will ensure that all drawing and specification information is consistent, that the scope of work is accurately defined, and that all of the necessary documentation is included.

1.1.4.5 Mechanical Drawings

All equipment/piping drawings (~50 total) have been completed, over two-thirds have been checked and over half have been back-checked and finalized. The companion instrumentation drawings (~30 total) are approximately 60% complete. Three drawings were developed and issued to the field which permit ongoing work on equipment placement setting:

- Leg extensions and load cell installation on M-G silicon bin.
- Access ladder on DCS distillation column.
- Nozzle additions and relocations on waste silica bin.

1.1.4.6 Electrical Design, Specification & Materials and

1.1.4.7 Electrical Drawings

Electrical design and drawing work was delayed pending completion of the mechanical design but was restarted in early March and will take approximately until mid-May to complete.

Class I-Division 2-Group B, Class I-Division 2-Group D, and unclassified electrical construction classification areas were reviewed and finalized with the safety engineer.

Design and routing for all electrical cable trays were completed. Routing of individual wire runs was initiated.

1.2 EQUIPMENT FABRICATION/DELIVERY

This report item includes all in-house and outside activities associated with fabrication, delivery, and vendor coordination for all items of equipment.

Many equipment items arrived at the EPSDU site, including:

- 469-14 - Instrument air compressor
- 469-20 - Instrument air dryer
- 466-04,05 - Cooling water pumps
- 453-02 - Agglomerator blower
- 458-04 - Silica drum packer
- 452-02 - Muriatic tailing column
- 426-02 - Quench contactor pump
- 436-02 - TCS distillate pump
- 436-04 - DCS distillate pump
- 436-05 - Spare chlorosilane pump
- 453-04,05 - Waste gas induction blowers
- 411-02 - M-G silicon storage bin
- 451-08 - Silica dust bin
- 451-18 - Caustic tank
- 461-02 - Hot oil expansion tank
- 461-06 - Diesel fuel day tank
- 464-06 - Thaw heater
- 456-08 - Tailing column pump
- 457-06 - Silica bag filter
- 429-02 - Quench & Solids venturi
- 459-16,20 - Agitators
- 642-02 - 480 V MCC No. 2
- 643-02 - Emergency generator
- 428-04 - Solids conveyor

The receipt of purchased items can be identified by consulting the equipment procurement status - see Appendix A.

1.2.8 Procurement Support

This task includes the Procurement Department effort necessary to initiate, monitor and control the purchase of equipment.

1.2.8.1 Purchasing, Expediting

The procurement status report is updated and issued monthly. The reporting data, including this quarter, is shown as Appendix A. Visits were

made to the fabricators of custom equipment items to assure the delivery of a quality product which conforms to specifications. Shelf type items are inspected upon receipt at the EPSDU site.

Inspection reports were prepared and issued for all equipment items which received one or more inspections during fabrication in the vendor shops. No problems were identified which have not been resolved in the shop or cannot be readily resolved in the field. These reports will be compiled and transmitted to JPL Q.A. personnel during a meeting at the EPSDU job site scheduled for April 2, 1981 to review both shop inspection procedures and field inspection/check-out procedures.

A material receiving log is being maintained which gives details of equipment received, date of receipt and site location (see Appendix A).

A tag number system is currently being developed and identification numbers will be added to the log.

1.2.8.2 Equipment Design and Inspection

The logic diagrams for the thermoflux heaters were reviewed and found to be missing key relays -- the vendor has agreed to retrofit each unit. A more complete logic diagram review is planned in the second quarter.

The electronic-grade cleaning of the silane column was observed. To check the effect of the cleaning procedure on the packing, a sample was taken and cleaned in the lab according to procedure. The procedure was completed and sample pieces were examined under a microscope together with some uncleaned control pieces. No significant change to the surface features was noticed. A small amount of metal had been eaten and the stainless surface was dulled. The fabricator was notified to continue, using the pre-established procedures.

A significant effort will be required to pre-clean piping and instrumentation components for either chlorosilane service (very clean) or silane service (ultra clean) prior to installation. This effort acknowledges the previously stated process design/start-up philosophy of minimizing all chances of introducing foreign contaminants into the system and, in turn, hopefully minimizing the start-up operating time required to make contaminant-free silane.

A lengthy listing was completed of all items to be cleaned at Tonawanda in May and June. During April, a similar listing will be completed of items to be cleaned at EPSDU during construction. Equipment used to clean components at Tonawanda will be relocated to the EPSDU job site in July, 1981.

1.2.8.4 Tonawanda Equipment Cleaning

Arrangements were made for the process group to receive a number of pieces at Tonawanda and control the cleaning of this equipment to proper standards. Equipment will be sent to a work area where it will be logged in, dismantled as necessary by the facility instrument shop technicians and welded as required by the facility fab shop. The instrument shop will clean, reassemble, leak test and calibrate under our supervision. The pieces will then be repackaged and logged out for shipment to the field.

1.3 INSTALLATION AND CHECKOUT

This report item includes all effort associated with award of subcontracts, providing instructions to the on-going subcontract activities, monitoring subcontractor performance, and checkout activities to ensure proper installation.

1.3.1 Installation Subcontracts

This task includes the effort for all installation activities assigned to subcontractors and includes labor, materials, and day-to-day job supervision.

Monthly reports are issued which include progress details, schedule and cost summaries.

1.3.1.2 Civil, Underground, Structural

The balance of the contractual work was completed in the first quarter. The pyrolysis/consolidation, control and water treatment buildings were completed. Yard work, underground piping and utilities were also finished. Figure 2, EPSDU site, is a photograph of the site taken in March which clearly shows the construction progress.

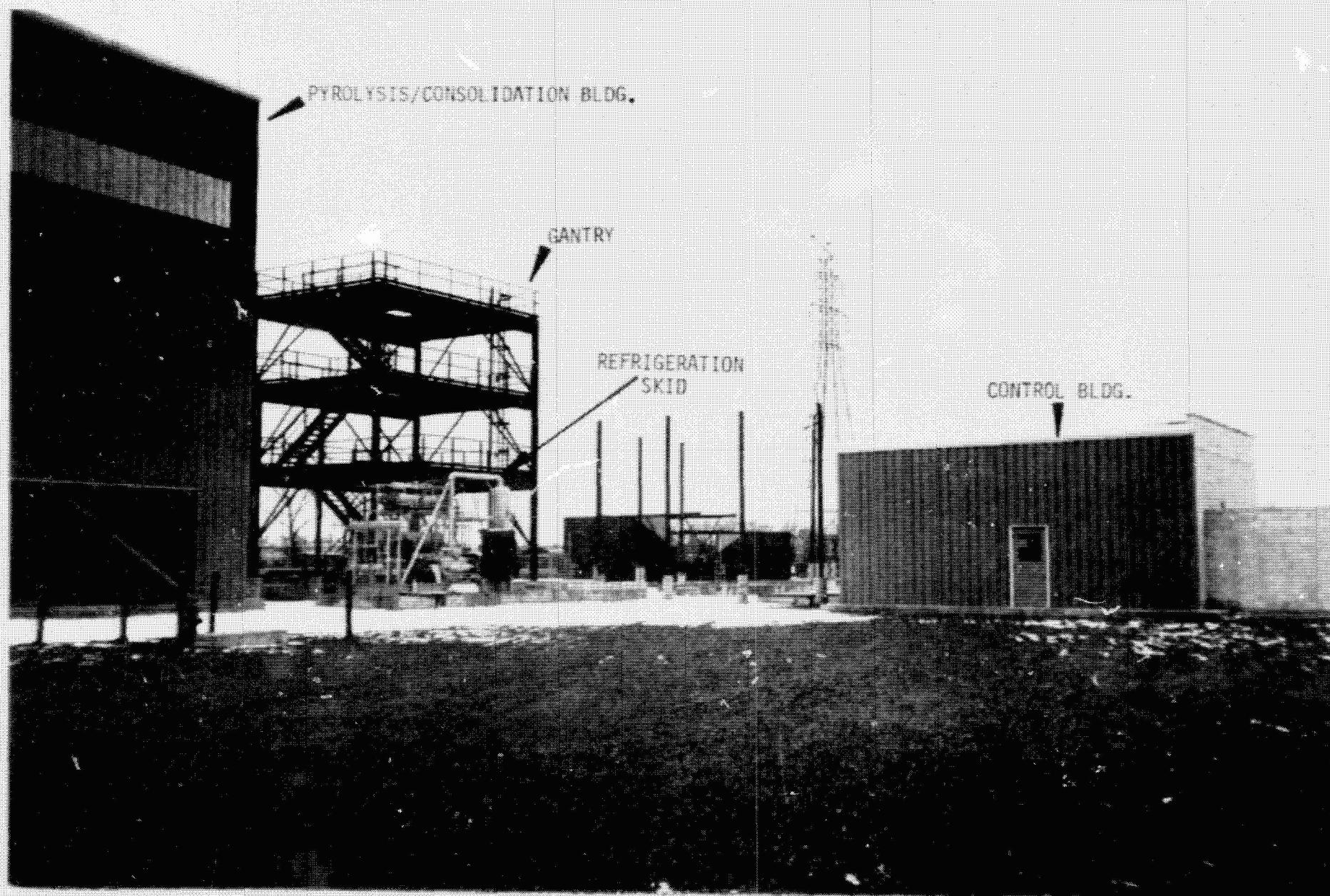


Figure 2. EPSDU Site

1.3.2 Installation Materials

Fifteen purchase orders were issued primarily for a total of 157 valves ranging from 1/4-inch to 1-inch sizes.

1.3.3 Installation Support

This task includes the in-house support associated with supervising, directing, and monitoring the installation effort.

1.3.3.1 Field Supervision

A resident construction superintendent for site plant construction is assigned to provide supervision of the contractors to assure that the work performed is in agreement with the facility plans.

Construction methods are observed to ascertain that the contractor operates in a safe and conscientious manner. He also visually checks process equipment as it is delivered on site to verify that no damage has occurred during shipment. A monthly construction report is prepared to record details of progress, schedule and cost.

The construction superintendent is supported by a clerk who handles and maintains documentation. A key, primary function is to maintain a 'Material Receiving Log', by which each item received onsite is registered detailing date received, purchase order, vendor, description, location and carrier. He will also be responsible for installing government tags on all capital items over \$1,000.

1.4 OPERATION

This work item includes all activities necessary to operate EPSDU to demonstrate operability, provide representative product samples, generate performance data for economic assessment and equipment and system design data for a commercial facility.

1.4.1 Preparation

This activity includes training of operating personnel, preparation of an operations manual and test plan.

The Plant Manager attended meetings at Tonawanda to complete the design safety review resolution report, finalize the liquid versus high pressure silane gas storage choice and to review projected 1.4 Operations costs with management.

The plant manager participated in the JPL/UCC bimonthly meeting held at Tonawanda on March 3rd through 7th. The EPSDU site was visited by the JPL review team along with UCC program management.

An Equal Employment Opportunity Program Affirmative Action Plan was prepared for EPSDU.

1.4.1.3 Operation Inspection & Calibration

It is planned that the Process Group will conduct on-site inspection of equipment items starting in April at East Chicago. The focus will be on equipment internals, since the plant manager is inspecting external details. The inspection will focus on personal safety and operability.

1.6 PROCESS SUPPORT R & D

The supporting R & D Program is separate from the mainstream design effort and includes all activities associated with analytical and experimental development of the free-space reactor, melting/consolidation system, fluid-bed reactor system, and quality control techniques and procedures. Information generated in this program will be used for the EPSDU effort and the commercial facility economic analysis.

1.6.1 Free-Space Reactor Development

This development task includes all experiments and analysis necessary to verify design data for the free-space pyrolysis reactor and to develop a new reactor model. This task was successfully completed and the accomplishments are summarized here.

1.6.1.1 PDU Experiments

OBJECTIVE

Construction and operation of the Tonawanda free-space reactor PDU had the objective of demonstrating long term steady-state operation, and developing a data base for use in scaling up to a larger size. Achieving these objectives would confirm the viability of the free-space reactor concept for EPSDU.

INTRODUCTION

Free-Space silane pyrolysis technology has been advanced significantly as a result of the JPL-sponsored development effort at Union Carbide. This activity builds on earlier UCC funded work involving the construction, operation and testing of a Free-Space Reactor Process Development Unit (FSR PDU). The testing at the Parma Technical Center demonstrated the feasibility of producing semi-conductor grade polycrystalline silicon by pyrolyzing high purity silane.

Under the current program a similar reactor with improved modifications was developed and tested at the UCC Tonawanda Facility. An initial series of test runs (7) indicated that long duration operation of the Free-Space Reactor was feasible provided that hard silicon deposits could be eliminated and the scraper seals and powder transport system improved.

These areas of concern directly impacted the design of the EPSDU Free-Space Reactor (FSR). This report summarizes the results of the experimental work in the second phase of Tonawanda's FSR development effort (Note that information concerning the runs during July and August of 1980 was documented in the Quarterly Progress Report, July-September 1980).

CONCLUSIONS

The experimental work resulted in the following conclusions which influence the design of the EPSDU Free-Space Reactor.

It was demonstrated that the occurrence of hard silicon deposits on the reactor wall could be eliminated by maintaining the peak reaction zone temperature (12 inches below the head) between 830^o and 900^oC. This was achieved by utilizing an induction heater coil with variable pitch windings (close pitch at each end and wider pitch at the center). The conversion of silane was in excess of 99.99%. Powder particle size and bulk density were reduced to 0.34 μm and 2.3 to 8.6 lb/ft³ from 0.90 μm and 9.4 to 16.9 lb/ft³ respectively, and is attributed to the higher reaction zone temperature.

New improved mechanical features — scraper seal, quartz liner support system and powder transport system — demonstrated feasibility and durability. All of these features will be used in the design of the EPSDU FSR.

Powder purity was not conclusively demonstrated due to the inadequacy of the sample preparation and analytical techniques used (AA, ICP). However, a polysilicon boule pulled from melted FSR powder was shown to have 55 Ω cm P-type resistivity.

In summary, the test program was very successful and provides a firm basis and understanding to confidently scale-up to the EPSDU design.

DISCUSSION

Test Runs

A total of twelve runs, Numbered 8 through 19, were made on the PDU during Phase II (See Table II). These were grouped into three campaigns during each of which the system was kept sealed and under inert gas purge between runs. Most of the major equipment changes were carried out between campaigns at which time the PDU was opened and thoroughly cleaned by vacuuming and washing with deionized water.

The first campaign included runs Number 8 through 13. Activity during this period centered on modifying the wall temperature profile and flow field in the reactor to eliminate the hard wall deposits. Before this campaign was started, the 5 cm thick ceramic wool blanket was extended to the top of the reactor shell to reduce heat losses and the induction coil was relocated to a higher position on the reactor.

Modifications made during the campaign included the installation of a new induction heater coil (with variable density windings) to improve the heat distribution within the reactor. Run Number 11 resulted in excessive temperature in the lower section of the reactor and this was corrected by removing a section of insulation blanket. Run Number 13 demonstrated a successful 12-hour duration run.

Reactor performance during this campaign was much improved over previous runs, silane conversion was better than 99.99% and no hard silicon deposits were detected on the reactor wall. Table II summarizes the results of the first campaign.

Following Run 13, the PDU was shutdown for two months for extensive modification. A new liner support system was fabricated and installed. This utilized Inconel springs to support the weight of the quartz liner, keep it in compression against the reactor head, and compensate for thermal expansion of the reactor. Sealing against the head was accomplished using Grafoil strips and the annular space between the liner and reactor was actively purged with argon introduced through ports machined in the reactor head.

TABLE II RUN SUMMARIES

<u>Run No.</u>	<u>Date</u>	<u>Duration (Hrs)</u>	<u>SiH₄ Rate (kg/hr)</u>	<u>Reaction Zone Temp (°C)</u>	<u>Powder Transferred (%)</u>	<u>Comments - Equipment Modification</u>
8	7/11/80	2.0	2.20	820	53	Old induction coil, 9.5cm below top, new insulation blanket, injection nozzle 10.2 cm into reactor. Simple liner support. No fluidizing cone. Bottom transfer.
9	7/22/80	2.0	2.27	840	69	As per Run No. 8
10	7/25/80	2.25	2.30	825	66	As per Run No. 8, but 2X absolute pressure (276 kPa vs 138 kPa). No effect on powder.
11	8/14/80	1.7	4.4/2.6 (3.64 av)	905	27	New induction coil (unequal spacing). 10.2 cm below top. Injection nozzle 14.0 cm into reactor. Lower coils overheated reactor.
12	8/18/80	3.1	4.5/3.4/2.2 (2.88 av)	870	45	Lower 18 cm of insulation blanket removed. Temperature in lower reactor reduced to acceptable level. Completion of milestone.
13	8/26/80	12.0	2.21	865	-	Completion of milestone. No equipment changes.

—END OF FIRST CAMPAIGN—

TABLE II RUN SUMMARIES (continued)

<u>Run No.</u>	<u>Date</u>	<u>Duration (Hrs)</u>	<u>SiH₄ Rate (kg/hr)</u>	<u>Reaction Zone Temp (°C)</u>	<u>Powder Transferred (%)</u>	<u>Comments - Equipment Modification</u>
14	10/23/80	3.0	2.30	865	75	New liner support and powder transport systems installed. Heating system unchanged. Powder stored under inert purge.
15	10/28/80	4.0	2.33	875	83	As in Run 14.
16	10/31/80	4.0	2.29	860	101	Completion of milestone. As in Run 14.
17	11/10/80	12.0	2.37	870	79	Completion of milestone. As in Run 14. 11.8 kg to Kayex.

—END OF SECOND CAMPAIGN—

18	12/22/80	12.0	2.4	880	57	New scraper installed. Other systems unchanged.
19	12/31/80	3.7	2.3	880	75	Consumption of last available silane (remaining cylinder pressures too low). Minimal effort to transfer powder.

—END OF THIRD (FINAL) CAMPAIGN—

A new powder transport system was also fabricated and installed. The arrangement is similar to that used to entrain material in blow-pot (batch) type pneumatic conveying systems. A truncated cone of porous media sat in the bottom of the hopper. Powder was removed through a vertical 3.8 cm pipe which exited the hopper through an unused port. Nitrogen could be supplied to the porous cone to aid powder flow. The space behind the cone was purged. Following the installation of the transport system, the PDU was cleaned with deionized water and readied for operation.

The second campaign encompassed Runs 14 through 17. These are summarized in Table II. No changes were made to the PDU during this series of runs, which were intended to test the performance of the liner support and powder transfer systems. PDU operation was extremely smooth throughout. All the runs were made at similar conditions of flow and had similar wall temperature profiles. During this campaign a serious effort was made to obtain an accurate mass balance for the PDU. The silane cylinders were weighed before and after each run, purge flows were checked and the recalibrated dry test meter was used to monitor the flow of gas out of the PDU.

Examination of the reactor liner following Run 17 showed that the new support system had operated as expected. No powder penetrated behind the liner and the reactor wall was completely undamaged. Deposition on the inside of the liner was limited to two bands of chemical vapor deposited (CVD) material, the upper band extending between 6 and 11 cm and the lower band extending between 55 and 60 cm below the reactor head. Scanning electron microscope (SEM) examination of the coating showed it to be less than 0.5 μm thick after 23 hours of operation. This represents an acceptable deposition rate for EPSDU operation.

The mass balance around the PDU for the second campaign was very good. The silicon balance was excellent and the hydrogen balance acceptable. A systematic error in estimating purge flows seems probable as the measured hydrogen flow exceeded the calculated value by approximately the same amount for each run.

Following the second campaign the PDU was shut down for installation of a new scraper/scraper seal assembly.

The new arrangement utilized an in-line double-ended air cylinder to minimize alignment problems, an elastometer wiper to minimize power intrusion along the shaft, and multiple O-ring seals with argon purge.

The third campaign Runs 18 and 19 completed the experimental program and is summarized in Table II. Run 18 was a long duration test intended to provide operating experience with the new scraper assembly. The run was very smooth.

Conveying of powder proceeded normally following each run. Run 19 was a medium duration test made primarily to use up the supply of silane remaining following Run 18. The run and subsequent powder conveying proceeded normally. The temperature profiles of both runs 18 and 19 responded as expected to the increased scraper stroke and reduced insulation blanket; wall temperatures in the lower reactor were reduced to acceptable levels.

Examination of the scraper following Run 19 showed that a small quantity of powder had penetrated past the elastomeric wiper and seal assembly. An improved cylinder and seal design will be used for EPSDU FSR which includes water cooling of the scraper shaft, use of a metallic lower wiper and use of elastomeric seals of different configuration for the secondary seal.

Analytical Results

Tests were run to determine a number of powder characteristics including levels of certain metallic impurities, bulk density, and powder specific surface area. The purity of the silane feed used was checked and the resistivity of a polysilicon rod pulled from melted powder determined. The results of these tests are summarized in Table III.

Bulk Density/Particle Size

Bulk density of the product powder varied widely throughout Phase II, but at $36 - 137 \text{ kg/m}^3$ ($2.3 - 8.6 \text{ lb/ft}^3$) was much lower than the $150 - 270 \text{ kg/m}^3$ ($9.4 - 16.9 \text{ lb/ft}^3$) found during Phase I for comparable silane feed rates.

This is probably attributable to the higher temperatures maintained in the upper reactor during Phase II which led to increased silane pyrolysis rates and apparently favored homogeneous nucleation over heterogeneous particle growth. This led to the production of finer powder and reduced the bulk density of the material.

The bulk densities measured during Phase II were sensitive to powder handling. A sample scooped from the top of the undisturbed powder bed in the hopper was of dramatically lower bulk density than any of the other Phase II samples, all of which were taken from storage drums following conveying of the powder.

Silane Feed Purity

Chromatographic analysis of the fresh silane supplied for use in Phase II showed less than 30 ppm total chlorosilanes, down from 1300 ppm in a cylinder used in Phase I. Volatile iron was down from 1.6 ppm to below the detection limit of the method used (30 ppb). Epitaxial films of greater than 160 Ω cm resistivity were obtained in Tonawanda using silane from the new supply. Films obtained from silane from the previous supply had shown resistivities on the order of 40 Ω cm or less. Thus, the silane used from Run 13 was of significantly higher quality than that used in previous runs.

Product Powder Purity

Product purity was evaluated in two ways: by direct analysis of the powder using atomic absorption (AA) and induction coupled plasma (ICP) spectrometry and by measurement of the resistivity of a polysilicon boule pulled from melted powder.

Typical analytical results are shown in Table III. The metallic contaminants were generally in the low ppm level. The levels of impurities found in the high purity control were generally comparable to those found in the powder.

Measurements of low concentration of metals in silicon is not an easy matter, and the sample preparation and analysis techniques available

during this program simply appear to have been inadequate. It seems likely that the powder was less contaminated than these results indicate. However, it is clear that considerable development work must be done before powder purity can be routinely determined by spectroscopy.

The resistivity measurement obtained on a polycrystalline ingot pulled from melted powder produced an excellent value of 55 Ω cm p-type essentially throughout a 7.5 cm diameter by 7.5 cm long polysilicon boule.

TABLE III

ANALYTICAL RESULTS (ppm ~ mass)

	Typical Sample	Control*	Detection Limit	
			AA	ICP
Fe	0.6	0.75	0.1	0.5
Cr	0.1	<0.1	0.1	0.4
Ni	0.3	0.15	0.15	0.4
Mn	0.1	0.1	0.05	0.1
Cu	0.2	0.2	0.1	0.4
Zn	0.5	0.15	0.02	0.2

*Dow Corning Poly. B \leq 0.3 ppb donor \leq 1.5 ppb
Surface etched before analysis.

Polycrystalline Silicon Resistivity: 55 Ω cm, P-type

X-Ray diffraction analysis: crystalline silicon powder

1.6.1.1.8 Reactor Scale-up and Design

A 100 MT/year free-space reactor/silicon hopper assembly for EPSPDU was designed based on the experience gained in operation of the PDU during the later half of 1980 (and theoretical support analyses).

The design consists of a quartz-lined Incoloy reactor, resistance heating furnace, and stainless steel product hopper. Detailed design drawings for fabrication of the equipment were prepared, and the calculations were documented in a separate design calculation package. The reactor and hopper were designed in accordance with established engineering practice for

pressure vessels with ASME code formulae serving as a design basis. Both the reactor and the hopper were designed for a maximum allowable working pressure (MAWP) of 14.0 psig with design temperatures of 1800°F for the reactor shell, 600°F for the reactor head, and 950°F for the hopper. Additionally, both the reactor and hopper are capable of withstanding full vacuum.

The reactor consists of a cylindrical Incoloy 800H shell, an 18-inch I.D. quartz liner, and a water-cooled stainless steel top head. The hopper is constructed of 304 stainless steel and consists of a torispherical upper head, a 48-inch O.D. cylindrical main shell section with an 18-inch access manway, and a Dynapore (porous 304 stainless steel) lined lower conical shell.

Silane gas is injected into the quartz-lined reaction chamber via a water-cooled injector tube which passes through the reactor head. The optimum temperature profile in the reactor is maintained by an externally mounted 10-zone split-tube furnace (resistance heaters) with provisions for independent (individual or multi-zone) control. The furnace is supported independently of the reactor/hopper vessel by two I-beams which attach to an external framework.

During the reaction process, silane gas (SiH_4) is decomposed to form silicon powder (Si) and hydrogen gas (H_2). The reaction products enter the hopper via a 20-inch communicating chamber in the upper head of the hopper. The silicon powder collects in the lower conical and straight shell sections of the hopper.

A manifold, consisting of eighteen sintered metal filter elements, is attached internally to the upper head of the hopper with a 2-inch pipe size nozzle passing through the head. Hydrogen gas from the reaction is drawn from the hopper through the filters to a separate external recovery system. The hopper has a net product volume of 79 cubic feet to a point 12 inches below the filters.

A product withdrawal line (1-1/2 inch diameter pipe) passes through the main shell of the hopper and terminates near the bottom of the conical shell. Hydrogen gas is injected from an external source through the lower cone

flange, into the annular space between the outer shell and the Dynapore (porous stainless) inner cone. The flow of gas facilitates pneumatic transfer of the powder from the hopper through the product withdrawal line. Powder flow is assisted by the flow of hydrogen gas through the sintered metal cone, and by an internal "breaker bar" mechanism mounted through the main shell immediately above the conical shell juncture. The "breaker bar" mechanism consists of 0.5-inch stainless steel rods (adjustable) mounted to a 1-inch stainless steel shaft which is driven by an externally mounted pneumatic rotary torque actuator to break up powder "bridging" in the hopper shell and cone.

Powder deposits on the quartz reactor chamber walls are removed by a stainless steel "scraper" ring capable of traversing a 40-inch length of the reactor chamber. The scraper is mounted on the end of the water-cooled shaft of a specially designed (Carter Controls) pneumatic cylinder suitable for high temperature operation. The pneumatic cylinder is externally attached to the top of the reactor head. The cylinder shaft passes through the water-cooled reactor head which contains an additional scraper and sweep purge chamber to protect and insure proper functioning of the pneumatic cylinder.

Cooling for the hopper is provided by clamp-on Panelcoil elements attached to the cylindrical shell of the hopper and by 0.5-inch O.D. copper tubing coils on the upper hopper head. Additional copper cooling coils are attached to the lower portion of the reactor shell (below the furnace). Heat transfer to the cooling coils and panels is enhanced by the use of a heat transfer cement.

The reactor/hopper assembly is supported by four external lugs attached to the main shell of the hopper. The support lugs will in turn be bolted to the same external framework to which the independently supported furnace is attached.

Drawing number D-2160600 (Figure 3), shows the final assembly -- Free-Space Silicon Reactor/Silicon Hopper for the EPSDU facility.

1.6.2 Melting/Consolidation

The design and development effort necessary to obtain a reliable melter for EPSDU involves UCC and sub-contractor (Kayex) effort.

TO: THE DIRECTOR, FBI
FROM: SAC, NEW YORK
SUBJECT: [REDACTED]
[REDACTED]



FOLDOUT FRAME

[illegible]

REV	DATE	BY	APP'D	DESCRIPTION
1				

PRECEDING PAGE BLANK NOT FILMED

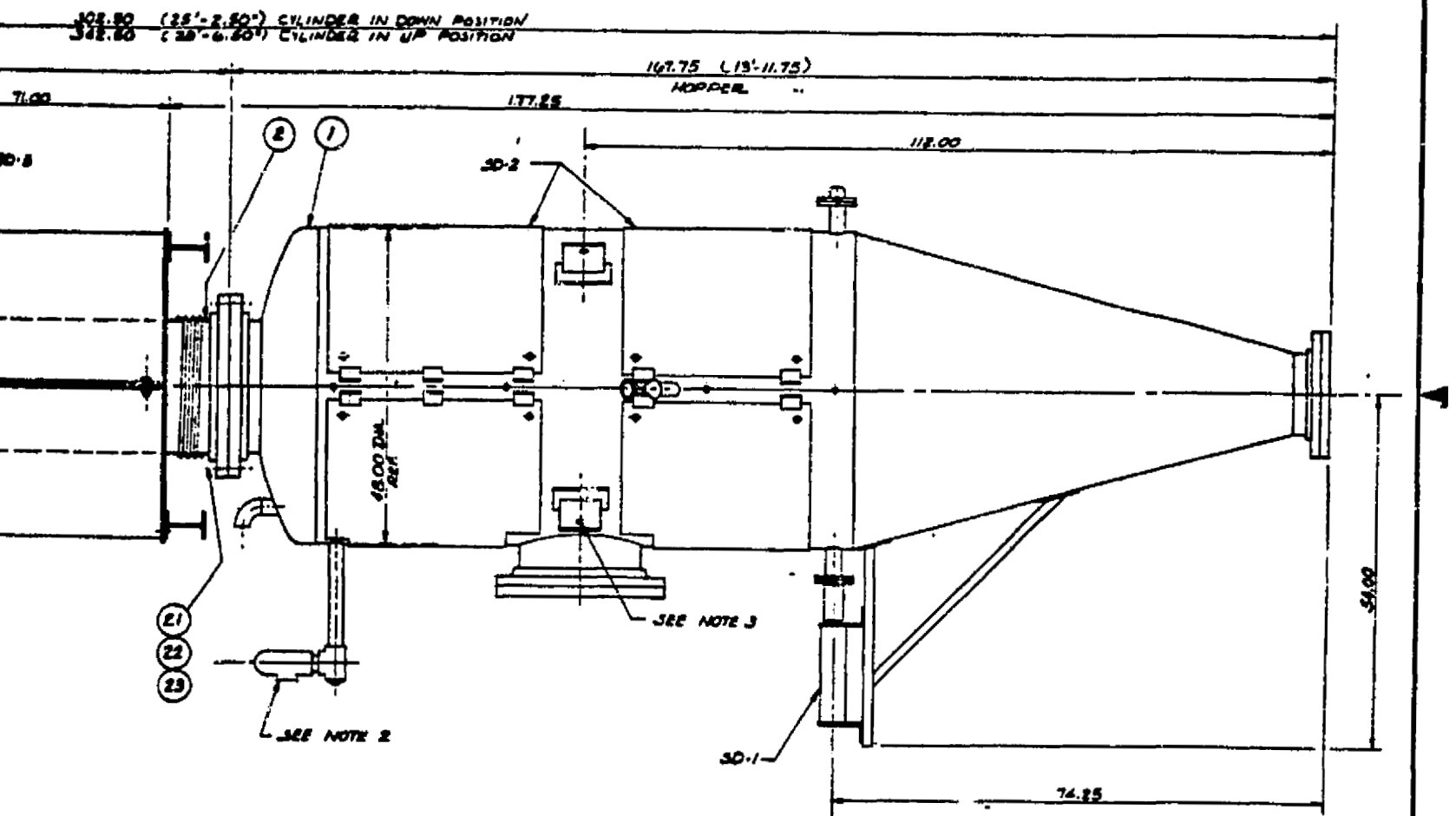


FIGURE 3

FOLDOUT FRAME 2

FOLDOUT FRAME
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FOR NOTES BILL OF MATERIAL & SDS
SEE BM-2160600

REFERENCE DRAWINGS
DESIGN CALCULATIONS A-2160638
CONNECTION LAYOUT C-2160643

WARNING MUST BE CLEANED PER LINDE
STANDARD SPEC. 303-1

TITLE: FINAL ASSEMBLY
FREE SPACE SILICON REACTOR /
SILICON HOPPER

UNION CARBIDE CORPORATION
LINDE DIVISION

DATE	BY	DATE	BY
10/1/60	WHE	10/1/60	WHE
CHECKED		APPROVED	
RJS		WHE	
D-2160600		D-2160600	

ESTIMATED WEIGHT

HOPPER & REACTOR ASSEMBLY - 5225 LBS
FURNACE ASSEMBLY - 4000 LBS

LIST ALTERATIONS ON BM-2160600 GR. I

ALTERATION	DATE	BY	APP'D	DESCRIPTION
------------	------	----	-------	-------------

1.6.2.1 Melter System Subcontract

The in-house effort involves establishing and managing the sub-contract and sub-contractor development, design, and fabrication efforts.

Kayex Corporation is developing the silicon melter system for EPSDU. The silicon consolidation scheme is based on melting the powder in a quartz crucible and dropping molten silicon shot from the crucible bottom into a cooling tower where the shot is solidified. The goal is to design and build a melting/consolidating system suitable for installation in the EPSDU.

The construction phase for the shotter system was much longer than planned due to difficulties in obtaining custom items made to the correct specifications. Initial checkout of equipment started in January. Difficulties were encountered in commissioning the induction heating system. The induction coil was twice returned to the factory to be reworked to obtain a satisfactory fitup.

The process development phase began on February 6th when the generator was finally commissioned. Five initial heating runs (without a melt) were conducted. These runs served to prove out each component during operation. One key problem encountered was that coil support brackets began melting even though radiation shields were in place. The coil supports were fabricated from Mykroy. During the fifth run (Feb. 27, 1981), 80% of the generator power was reached and the temperature in the susceptor rose to 1463°C at which time power was discontinued due to the feedthrough plate beginning to melt. Although the plate is mounted to a water-cooled section, cooling was insufficient. This problem will be corrected by using a higher temperature material.

A photograph of the melter/shotter system in place at Kayex is shown in Figure (4). Also shown is a view of the induction and susceptor (Figure 5). During the next quarter, the remaining system components will be checked out and modifications made to be able to have a controlled melt and produce silicon shot.

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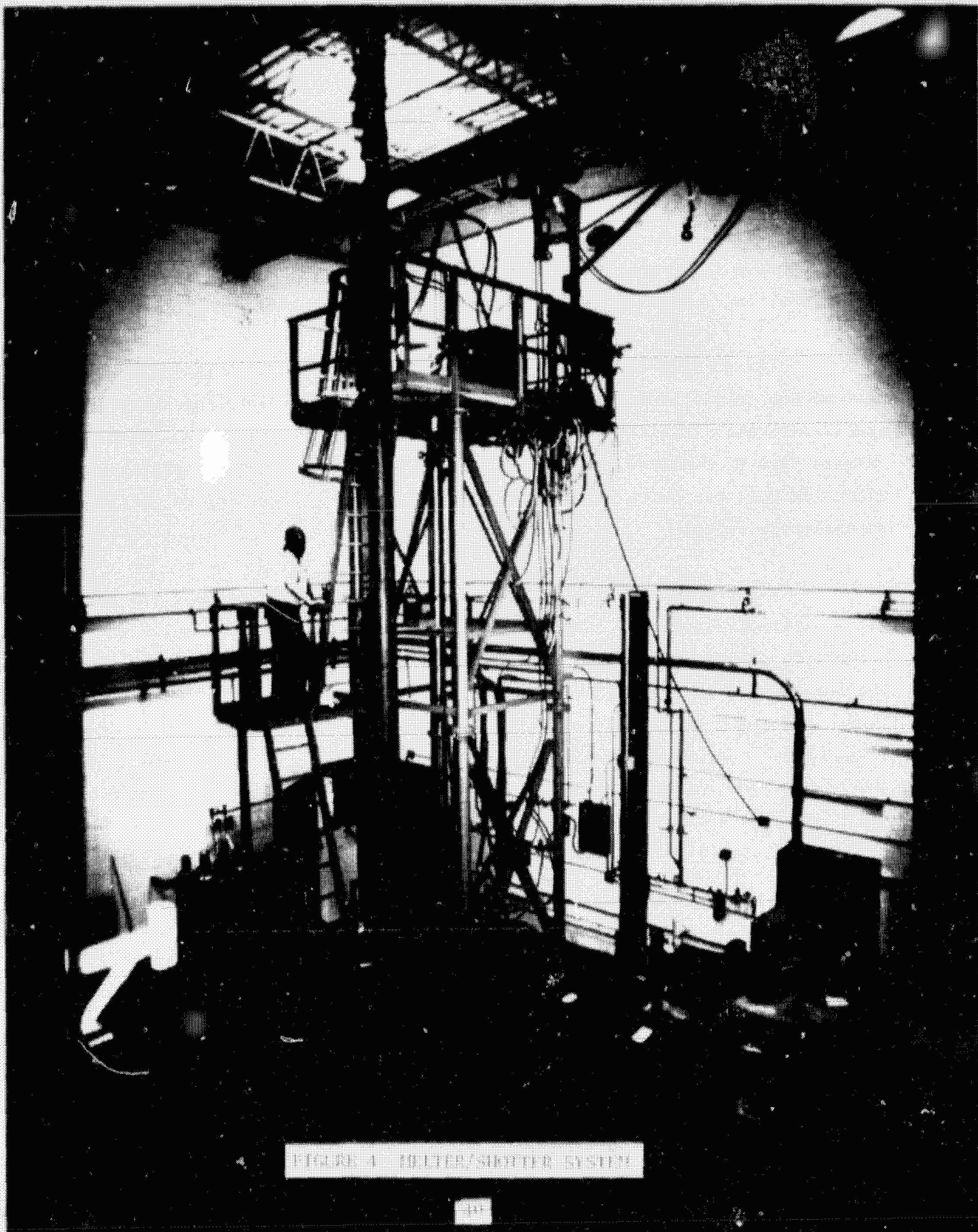


FIGURE 4. FILTER/SEPARATOR SYSTEM

10

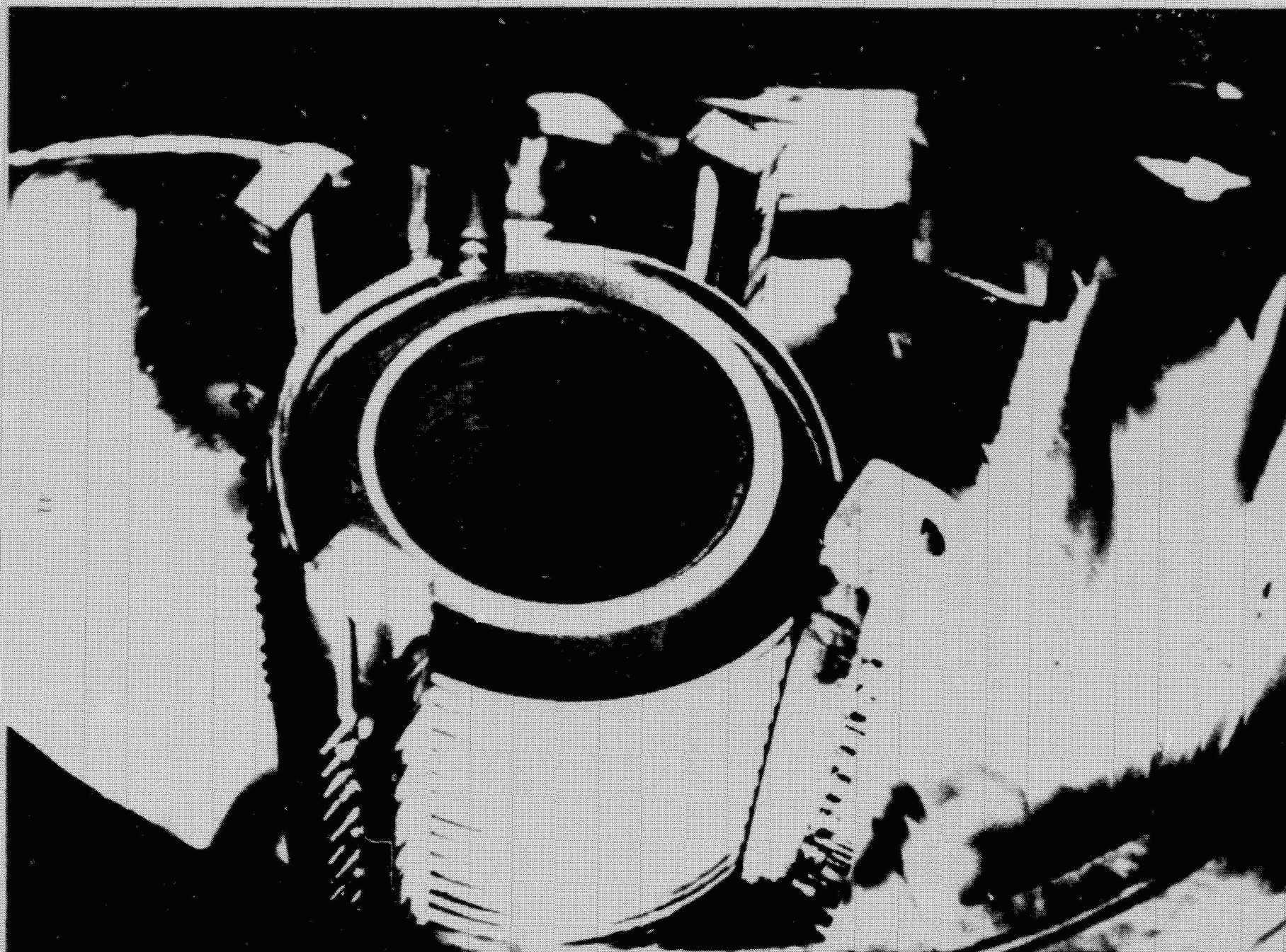


FIGURE 5 INDUCTION COIL AND SUSCEPTOR FOR METER/SHOOTER SYSTEM

1.6.3 Fluid-Bed Development

This development program includes all analytical, experimental, and design effort associated with developing a fluid-bed reactor as an alternative or backup system to the free-space reactor.

1.6.3.4 Fluid-Bed Pyrolysis (FDU)

This work item consists of all effort associated with the design, fabrication, and testing of an experimental unit to establish design data for an EPSDU-scale system.

The activity during this quarter was to complete the piping of the fluid-bed unit, check out system, install and calibrate instrumentation, calibrate controls and flow meters. Also, in support of the proposed experimental program, a computer program for data reduction was written.

Experimental data for each run will be stored on a ticker-tape by the data-logger. This information will be transferred to DTSS computer system for interpretation and generation of final data tables.

A safety review meeting was held with both the safety and engineering departments. The procedures of operation of the fluid-bed were reviewed and the system was inspected.

An experimental plan has been written for the fluid-bed pyrolysis program with schedules. During the next quarter, the fluid-bed reactor will be heated up to 650°C with resistance band heaters, and the experimental program will begin.

1.7 MANAGEMENT AND DELIVERABLES

This report item includes all activities associated with managing the program and insuring that all deliverables are made in accordance with the program requirements.

17th Progress Integration Meeting (PIM)

This meeting was held in early February at Pasadena. UCC was represented by J. H. Lorenz, W. G. Bancroft, and H. Morihara. Two presentations were made; one at the plenary session which showed the program status and the second at the Task I technical session.

The EPSDU gantry model was also on display at the meetings. A recommended revised program for FY1981 was outlined to accommodate the reduced funding but also minimize the EPSDU start-up schedule slippage.

Balance of Program Activities

UCC prepared a proposed activity and schedule plan based on the discussion at the February PIM and transmitted it on February 27, 1981. All available manpower was mobilized to prepare the documents in a three-week period. The proposed plan which was based on current thinking, labor and material rates, consisted of a technical volume and cost volume consistent with the reduced FY81 funding.

JPL/UCC Bimonthly Meeting

The meeting was held at Tonawanda during March 5th through March 11th. The meeting also combined with a fact-finding meeting and discussion of the documents previously submitted.

The schedule called for a full week of meetings, including weekend working sessions. The regular program and status reports were given and visits to Kayex and EPSDU completed the week. UCC submitted, at JPL's request, the revised program which describe our remaining technical work and our estimate-to-complete budgets. The cost volume included updated inflation factors, new work items deferred in 1979, and updated schedules and associated costs.

Our progress report indicated the EPSDU civil contract is essentially complete. The buildings are up and the roads are complete. Equipment is arriving and is being set in place or stored. The mechanical and electrical

contracts will be awarded in the late second quarter and early third quarter. All major equipment is on order. Design work is winding down, and the final P & I is issued and scale models are being updated. Designs are being completed for the pyrolysis and melter items.

On the R & D work, the reports on the free-space reactor and quality control procedure were reviewed. The fluid bed unit is checked out and will be ready to run (April to September 1981).

Quarterly Progress Review With Kayex

Kayex subcontract work is some 2 to 3 months behind schedule due to vendor problems with the melter unit. These are now being corrected and revised schedules are being considered to minimize slippage.

Backup Materials

Subsequent to the March Fact-Finding Meeting, a package of additional backup information was prepared and mailed to JPL to support, in more detail, the revised schedule and cost.

Deliverables

The monthly technical progress reports were issued along with the monthly financial and management reports.

The fourth quarter 1980 progress report was delayed but will be issued in April 1981.

Major Milestones

The major milestones reached this quarter are presented in Table IV.

TABLE IV MAJOR MILESTONES REACHED

<u>WBS NO.</u>	<u>MILESTONE</u>	<u>DESCRIPTION OF MAJOR MAILESONE</u>
1.1.3.1	K	P & I diagram issued and control panel RFR's issued March
1.2.1.1	E	Quarterly Procurement Status Report issued
1.2.1.2	E	Quarterly Procurement Status Report issued
1.2.1.4	E	Quarterly Procurement Status Report issued
1.2.2.1	H	Final Invoice payment for Power Supply
	E	Quarterly Procurement Status Report issued
1.2.2.3	G	All equipment received
	E	Final invoice payment
1.2.3.2	E	Quarterly Procurement Status Report issued
	G	All equipment received
1.2.3.3	E	Quarterly Procurement Status Report issued
	G	All equipment received
1.2.4.1	E	Quarterly Procurement Status Report issued
	G	All equipment received
1.2.4.2	E	Quarterly Procurement Status Report issued
1.2.4.3	E	Quarterly Procurement Status Report issued
1.2.4.4	E	Quarterly Procurement Status Report issued
1.2.4.5	E	Quarterly Procurement Status Report issued
	G	All equipment received
1.2.4.6	E	Quarterly Procurement Status Report issued
1.2.4.7	E	Quarterly Procurement Status Report issued
1.2.5.1	G	All equipment received
1.2.5.2	G	All equipment received
1.2.5.3	G	All equipment received
	H	Final invoice payment

TABLE IV MAJOR MILESTONES REACHED (continued)

<u>WBS NO.</u>	<u>MILESTONE</u>	<u>DESCRIPTION OF MAJOR MILESTONE</u>
1.2.5.4	E	Quarterly Procurement Status Report issued
	G	All equipment received
1.2.5.6	E	Quarterly Procurement Status Report issued
1.2.6.1	E	Quarterly Procurement Status Report issued
1.3.1.2	F	Completed civil work
1.3.1.3	J	Completed structural work
1.6.2.1.4	C	Interim report draft issued
1.7.3	N,P,Q	Monthly Financial & Management Reports issued
	N,P,Q	Monthly Technical Progress Reports issued
1.7.3	G,H	Bi-Monthly Review Meetings
	C	Program Integration Meeting
	K	Engineering Design Package
	L	Installation Drawing Package
	M	Interim Report on Sub-contract
	N	Slim-Rod Reactor Report
	P	Silane Analysis - Epitaxy

SECTION III. CONCLUSIONS

Significant highlights and conclusions are presented according to the relevant WBS numbers.

1.1 DESIGN/PROCUREMENT

1.1.1 Process Design

- Specifications for valves were written and valves were ordered.
- Operation logic for startup/shutdown was agreed upon with the Instruments and Controls Group.
- A cleaning procedure was established for cleaning of component used in chlorosilane streams.
- Preliminary process flow diagram for pyrolysis was prepared.

1.1.2 Facility Design

- Safety review team recommends liquid phase storage for silane at EPSDU.
- The air discharge permit has been approved by the Indiana Air Quality Office.

1.1.3 Equipment Design, Specifications, Procurement

- Revision "G" of P & I diagram was issued and is regarded as the final version.
- A custom stack gas analyzer is being designed to meet EPSDU requirements.

1.1.4 Installation Design, Specification, Procurement

- Procedural specifications for cleaning silane piping was completed.
- Equipment/Piping drawings are complete and are under review.

1.2 EQUIPMENT FABRICATION/DELIVERY

- Fifty-five additional purchase orders were issued.
- Many major pieces of equipment were received on site.
- Visits were made to fabricators of key components to conduct inspections.
- A material receiving log is being maintained.

1.3 INSTALLATION & CHECKOUT

- Civil, underground and structural work was completed.
- Purchase orders for additional valves were issued.

1.6 PROCESS SUPPORT R&D

1.6.1 Free-Space Reactor Development

- Summary of Free-Space Reactor Development issued.

1.6.2 Melting/Consolidation

- Construction phase complete.
- Melter/Shotter system is being checked out.

1.6.3 Fluid-Bed Development

- Safety review was conducted.
- Data reduction program was written.
- System was instrumented and ready for start-up.

SECTION IV. PROJECTED QUARTERLY ACTIVITIES

1.1 EF&DU DESIGN/PROCUREMENT

1.1.1 Process Design

- Process Design Group will continue to communicate and review designs with Engineering to insure that all requirements are being met.
- Pyrolysis/Melting Process Package will be reviewed and issued to Engineering.
- Equipment functional specifications for pyrolysis will be completed.

1.1.2 Facility Design

- Modification to the gantry scale model will begin.
- Continue facility scale model construction.

1.1.3 Equipment Design, Specification, Procurement

- The preliminary Control Logic Diagram will be completed in April and finalized after review in May.
- Continue development of data collection system programming.

1.1.4 Installation Design, Specification, Subcontract

- Chlorosilane piping cleaning specifications will be finalized.
- Field Bill of Materials document will be completed.
- Requisitioning piping components and materials will be completed.
- Mechanical bid package will be reviewed and issued.
- Electrical design activity will continue.

1.2 EQUIPMENT FABRICATION/DELIVERY

- JPL, Quality Assurance personnel will meet at EPSDU site to review inspection procedures.
- The material/equipment received at the EPSDU site will be logged and tagged.
- An area will be prepared at Tonawanda for cleaning specialty items.

1.3 INSTALLATION & CHECKOUT

- Equipment received will be inspected.
- Field progress reports will be issued monthly.
- Equipment delivered to the site will be placed on foundations or stored.

1.4 OPERATION

- The plant manager will visit Keasby, N.J., Specialty Gas Plant to observe silane production procedures.
- He will also give assistance for equipment inspection.
- Operations Manual preparation will start.

1.5 COMMERCIAL PROCESS ECONOMIC ANALYSIS

- No activity is planned.

1.6 PROCESS SUPPORT R&D

1.6.2 Melting/Consolidation

- Melting/shotting runs will be conducted using chunk silicon.

1.6.3 Fluid-Bed Development

- The fluid-bed PDU will be started up using acid-washed silicon and hydrogen for fluidization.
- Band heaters will be used to provide a uniform bed temperature.

1.7 MANAGEMENT AND DELIVERABLES

- Monthly Financial Reports will be issued.
- First Quarterly Progress Report (Jan - Mar 1981) will be issued.
- An update of PMS will be started to incorporate cost and schedule changes.

APPENDIX A

EQUIPMENT PROCUREMENT STATUS

MARCH 1981

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	DWG REC'D	EQUIPMENT REC'D
453-04-05 Waste Gas Induction Blower	50001	✓	✓	✓	✓	✓	✓	✓
453-02 Agglomeration Blower	50002	✓	✓	✓	✓	✓	✓	✓
	50003							
	50004							
426-02 Quench Contactor Pump	50005	✓	✓	✓	✓	✓	✓	✓
	50006							
	50007							
	50008							
	50009							
423-02, 03 Recycle H ₂ Compressor	50010	✓	✓	✓	✓	✓	✓	
443-02 Pyrolysis H ₂ Compressor	50011	✓	✓	✓	✓	✓	✓	
466-02 Hot Oil Pump	50012	✓	✓	✓	✓	✓	✓	✓
466-04, 05 Cooling Water Pump	50013	✓	✓	✓	✓	✓	✓	
424-02 Quench Condenser	50014	✓	✓	✓	✓	✓	✓	✓
424-04 434-06, 10 Reboilers	50015	✓	✓	✓	✓	✓	✓	✓

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	CERTIFIED DNG REC'D	EQUIP-MENT REC'D
434-02 Stripper Condenser	50016	✓	✓	✓	✓	✓	✓	✓
421-12, 16 441-06 Tanks	50017	✓	✓	✓	✓	✓	✓	✓
464-02, 04 Ventilation Heat Exchangers	50018							
434-08, 14, 18 Column Condensers	50019	✓	✓	✓	✓	✓	✓	✓
	50020							
	50021							
434-12, 16, 24 444-02 Coolers	50022	✓	✓	✓	✓	✓	✓	
	50023							
	50024							
	50025							
	50026							
	50027							
434-26 Refrig. Heating Coil	50028							
	50029							
	50030							

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	CERTIFIED DWG REC'D	EQUIPMENT REC'D
	50031							
	50032							
411-02, 441-04 461-08 Bins	50033	✓	✓	✓	✓	✓	✓	✓
	50034							
421-02,04,06,08,10,14,18 451-04,06 Tanks	50035	✓	✓	✓	✓	✓	✓	✓
	50036							
	50037							
	50038							
	50039							
431-04, 06, 08, 10 435-02, 04 Tanks & Reactors	50040	✓	✓	✓	✓	✓	✓	✓
	50041							
	50042							
	50043							
	50044							
	50045							

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	CERTIFIED DWG REC'D	EQUIPMENT REC'D
	50046							
	50047							
	50048							
	50049							
	50050							
451-10 Waste Neutralizer	50051							
	50052							
	50053							
	50054							
461-02 Hot Oil Expansion Tank	50055	✓	✓	✓	✓	✓	✓	✓
425-02 Hydrogenation Reactor	50056	✓	✓	✓	✓	✓	✓	
445-02 Quartz Liner	50057							
	50058							
445-02 Pyrolysis Reactor and Hopper	50059							
417-02 457-04, 06 Filters	50060	✓	✓	✓	✓	✓	✓	✓

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	CERTIFIED DWG REC'D	EQUIPMENT REC'D
427-02 Crude TCS Filter	50061	✓	✓	✓	✓	✓	✓	✓
437-02 Silane Ultra Filter	50062	✓	✓	✓	✓	✓	✓	✓
	50063							
	50064							
	50065							
	50066							
448-08, 10 Loading Scales	50067							
	50068							
448-04 Boule Cart	50069							
448-14 Boule Scale	50070							
	50071							
	50072							
458-04 Silica Drum Packer	50073	✓	✓	✓	✓	✓	✓	✓
	50074							

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	CERTIFIED DWG REC'D	EQUIPMENT REC'D
459-02, 04 Venturi and Scrubber	50075	✓	✓	✓	✓	✓	✓	✓
429-04 Superheater	50076	✓	✓	✓	✓	✓	✓	✓
	50077							
	50078							
	50079							
	50080							
	50081							
	50082							
459-08, 10, 12, 14 Waste Burners	50083	✓	✓	✓	✓	✓		✓
	50084							
454-04 Silica Agglomerator	50085	✓	✓	✓	✓	✓	✓	
469-02 Cooling Tower	50086	✓	✓	✓	✓	✓	✓	✓
469-06 Cooling Tower Treatment	50087	✓	✓	✓	✓	✓	✓	✓
469-12 Refrigeration System	50088	✓	✓	✓	✓	✓	✓	✓

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	CERTIFIED DWG REC'D	EQUIPMENT REC'D
469-16 Therminol Heater	50089	✓	✓	✓	✓	✓	✓	✓
452-02 Muriatic Tailing Column	50090	✓	✓	✓	✓	✓		
456-08 Tailing Column Pump	50091	✓	✓	✓	✓	✓	✓	✓
469-14 Instrument Air Package	50092	✓	✓	✓	✓	✓	✓	✓
642-02 MCC	50093	✓	✓	✓	✓	✓		✓
641-02 Transformer	50094	✓	✓	✓	✓	✓	✓	✓
	50095							
365-02 Quality Control Trailer	50096	✓	✓	✓	✓	✓		
	50097							
411-08 TL Argon Tank	50098							
461-04, 06 Fuel Oil Storage Tank	50099	✓	✓	✓	✓	✓		✓
	50100							
	50101							
643 Emergency Generator	50102	✓	✓	✓	✓	✓		✓

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	CERTIFIED DWG REC'D	EQUIPMENT REC'D
463-06, 08 Ventilation Blower	50103							
	50104							
426-06, 07 STC Pumps	50105	✓	✓	✓	✓	✓	✓	
	50106							
For 432-08 Internals for Silane Column	50107	✓	✓	✓	✓	✓	✓	✓
Chlorosilane Analysis	50108	✓	✓	✓	✓	✓		
UV Spectrophotometer	50109	✓	✓	✓	✓	✓		✓
Elemental Analysis	50110	✓	✓	✓	✓	✓		✓
Silicon Melting Furnace	50111	✓	✓	✓	✓	✓		✓
432-02 Stripper Column	50112	✓	✓	✓	✓	✓		✓
432-04 TCS Column	50113	✓	✓	✓	✓	✓		✓
432-06 DCS Column	50114	✓	✓	✓	✓	✓		✓
459-16 20 Agitator	50115	✓	✓	✓	✓	✓	✓	✓
429-02 Quench & Solids Removal Contractor	50116	✓	✓	✓	✓	✓	✓	✓

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	CERTIFIED DWG REC'D	EQUIPMENT REC'D
	50117							
	50118							
466-06, 07 Fuel Oil Pumps	50119	✓	✓	✓	✓	✓	✓	✓
	50120							
	50121							
432-08 Silane Column	50122	✓	✓	✓	✓	✓		✓
	50123							
Field Instrumentation	50124	✓	✓	✓	✓	✓		
469-20 Instrument Air Dryer	50125	✓	✓	✓	✓	✓	✓	✓
428-04 Solids Conveyor	50126	✓	✓	✓	✓	✓	✓	✓
	50127							
	50128							
464-06 Thaw Heater	50129	✓	✓	✓	✓	✓	✓	✓
Program Controller	50130	✓	✓	✓	✓	✓		

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	CERTIFIED DNG REC'D	EQUIP-MENT REC'D
7 Haskel Pumps	50131	✓	✓	✓	✓	✓		
Pressure Lubricators	50132	✓	✓	✓	✓	✓		✓
8 BP Valves & 7 Springs	50133	✓	✓	✓	✓	✓		✓
Main Instrument Panel "A"	50134							
Printer	50135	✓	✓	✓	✓	✓		
Computer	50136	✓	✓	✓	✓	✓		
Memorex Cartridges	50137	✓	✓	✓	✓	✓		
Automatic Valves	50138	✓	✓	✓	✓	✓		
Automatic Valves	50139	✓	✓	✓	✓	✓		✓
Automatic Valves	50140	✓	✓	✓	✓	✓		✓
Automatic Valves	50141	✓	✓	✓	✓	✓		✓
Automatic Valves	50142	✓	✓	✓	✓	✓		✓
	50143							
	50144							
7 Gauges	50145	✓	✓	✓	✓	✓		✓

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	CERTIFIED DWG REC'D	EQUIP-MENT REC'D
Pressure Lubricators	50146	✓	✓	✓	✓	✓		✓
Thermocouples	50147	✓	✓	✓	✓	✓		
7 Level Switches	50148	✓	✓	✓	✓	✓		
	50149							
Sewer Tie-In Line	50150	✓	✓	✓	✓	✓		
Service Agreement	50151	✓	✓	✓	✓	✓		
	50152							
Lever Transmitters & Switches	50153	✓	✓	✓	✓	✓		✓
Field Instrumentation	50154	✓	✓	✓	✓	✓		
Conditioners & Process Filters	50155	✓	✓	✓	✓	✓		
sample Conditioners	50156	✓	✓	✓	✓	✓		
Sample Conditioners	50157	✓	✓	✓	✓	✓		
	50158							
	50159							
Caustic Storage Tank	50160	✓	✓	✓	✓	✓		✓

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	DWG REC'D	EQUIPMENT REC'D
Immersion Heater	50161	✓	✓	✓	✓	✓	✓	
Fuel Tank	50162	✓	✓	✓	✓	✓	✓	✓
Shipping Preparation & Freight	50163					✓		
Automatic Valve	50164	✓	✓	✓	✓	✓		
	50165							
Main Instrument Panel "A"	50166	✓	✓	✓	✓	✓		
Main Instrument Panel "A"	50167	✓	✓	✓	✓	✓		
Field Instrumentation	50168	✓	✓	✓	✓	✓		
Main Instrument Panel "A"	50169	✓	✓	✓	✓	✓		
Main Instrument Panel "A"	50170	✓	✓	✓	✓	✓		
	50171							
Main Instrument Panel "A"	50172							
Main Instrument Panel "A"	50173	✓	✓	✓	✓	✓		
Main Instrument Panel "A"	50174							
Main Instrument Panel "A"	50175	✓	✓	✓	✓	✓		

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	DWG REC'D	EQUIPMENT REC'D
Main Instrument Panel "A"	50176	✓	✓	✓	✓	✓		
Field Instrumentation	50177	✓	✓	✓	✓	✓		✓
Field Instrumentation	50178	✓	✓	✓	✓	✓		✓
Field Instrumentation	50179	✓	✓	✓	✓	✓		✓
Field Instrumentation	50180	✓	✓	✓	✓	✓		
Field Instrumentation	50181	✓	✓	✓	✓	✓		✓
Field Instrumentation	50182	✓	✓	✓	✓	✓		✓
Field Instrumentation	50183	✓	✓	✓	✓	✓		✓
Field Instrumentation	50184	✓	✓	✓	✓	✓		✓
Field Instrumentation	50185	✓	✓	✓	✓	✓		
	50186							
Filter Bags	50187	✓	✓	✓	✓	✓	✓	
Valves	50188	✓	✓	✓	✓	✓		✓
Valves	50189	✓	✓	✓	✓	✓		✓
Automatic Valves	50190	✓	✓	✓	✓	✓		✓

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	DWG REC'D	EQUIP-MENT REC'D
Automatic Valves	50191	✓	✓	✓	✓	✓		
Automatic Valves	50192	✓	✓	✓	✓	✓		✓
Automatic Valves	50193	✓	✓	✓	✓	✓		✓
Check Valves	50194	✓	✓	✓	✓	✓		✓
	50195							
	50196							
Field Instrumentation	50197	✓	✓	✓	✓	✓		✓
	50198							
Field Instrumentation	50199	✓	✓	✓	✓	✓		
Field Instrumentation	50200	✓	✓	✓	✓	✓		
Field Instrumentation	50201	✓	✓	✓	✓	✓		✓
Controller	50202	✓	✓	✓	✓	✓		✓
	50203							
Instrument Field List	50204	✓	✓	✓	✓	✓		✓
Instrument Field List	50205	✓	✓	✓	✓	✓		✓

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	DWG REC'D	EQUIP-MENT REC'D
Manual Valve Packless	50206	✓	✓	✓	✓	✓		
Rubber Hose	50207					✓		✓
	50208							
	50209							
Field Instrumentation	50210	✓	✓	✓	✓	✓		✓
Field Instrumentation	50211	✓	✓	✓	✓	✓		✓
Field Instrumentation	50212	✓	✓	✓	✓	✓		✓
	50213							
Field Instrumentation	50214	✓	✓	✓	✓	✓		✓
Field Instrumentation	50215	✓	✓	✓	✓	✓		✓
Manual Valves "Packless"	50216	✓	✓	✓	✓	✓		
	50217							
Main Instrument Panel	50218	✓	✓	✓	✓	✓		
	50219							
	50220							

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	DWG REC'D	EQUIP-MENT REC'D
	50221							
	50222							
	50223							
Rav Valve Packing Pressurization	50224	✓	✓	✓	✓	✓		
Solenoid Valves	50225	✓	✓	✓	✓	✓		
Teflon-Lined Piping	50226	✓	✓	✓	✓	✓		
Sample Conditioners	50227	✓	✓	✓	✓	✓		
Sample Conditioners	50228	✓	✓	✓	✓	✓		
Fittings	50229	✓	✓	✓	✓	✓		
48 Packless Valves & 18 Diaphragms	50230	✓	✓	✓	✓	✓		
Sample Conditioners	50231	✓	✓	✓	✓	✓		
Manual Knife Valve	50232	✓	✓	✓	✓	✓		
	50233							
	50234							
	50235							

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EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	DWG REC'D	EQUIPMENT REC'D
SS Tubing	50236					✓		
	50237							
High Temperature Check Valves	50238	✓	✓	✓	✓	✓		
High Temperature Manual Globe Valves	50239	✓	✓	✓	✓	✓		
Manual Valves	50240	✓	✓	✓	✓	✓		
Expansion Joints	50241	✓	✓	✓	✓	✓		
Expansion Joints	50242	✓	✓	✓	✓	✓		
Manual 3-Way Valves	50243	✓	✓	✓	✓	✓		
Pump Connectors	50244	✓	✓	✓	✓	✓		✓
Diaphragm Pump	50245	✓	✓	✓	✓	✓		
Automatic Valve	50246	✓	✓	✓	✓	✓		
	50247							
	50248							
	50249							

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	DWG REC'D	EQUIP- MENT REC'D
	50250							
Field Instrumentation	50251	✓	✓	✓	✓	✓		
Field Instrumentation	50252	✓	✓	✓	✓	✓		
Field Instrumentation	50253	✓	✓	✓	✓	✓		
Field Instrumentation	50254	✓	✓	✓	✓	✓		
Field Instrumentation	50255	✓	✓	✓	✓	✓		
Field Instrumentation	50256	✓	✓	✓	✓	✓		
	50257							
Field Instrumentation	50258	✓	✓	✓	✓	✓		
Field Instrumentation	50259	✓	✓	✓	✓	✓		
	50260							
	50261							
	50262							
	50263							
	50264							

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFA ISSUED	P.O. ISSUED	DWG REC'D	EQUIP-MENT REC'D
	50265							
Field Instrumentation	50266	✓	✓	✓	✓	✓		
	50267							
	50268							
	50269							
	50270							
	50271							
Sample Conditioners	50272	✓	✓	✓	✓	✓		
	50273							
HCl Monitor	50274	✓	✓	✓	✓	✓		
HCl Monitor	50275	✓	✓	✓	✓	✓		
HCl Monitor	50276	✓	✓	✓	✓	✓		
HCl Monitor	50277	✓	✓	✓	✓	✓		
Thermostat & Finstrip Heater	50278	✓	✓	✓	✓	✓		
HCl Monitor	50279	✓	✓	✓	✓	✓		

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EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	DWG REC'D	EQUIPMENT REC'D
HCI Monitor	50280	✓	✓	✓	✓	✓		
HCI Monitor	50281	✓	✓	✓	✓	✓		
	50282							
Main Instrument Panel "A"	50283	✓	✓	✓	✓	✓		
	50284							
	50285							
Main Instrument Panel "A"	50286	✓	✓	✓	✓	✓		
	50287							
Main Instrument Panel "A"	50288	✓	✓	✓	✓	✓		
Main Instrument Panel "A"	50289	✓	✓	✓	✓	✓		
	50290							
	50291							
	50292							
	50293							
	50294							

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	DWG REC'D	EQUIP- MENT REC'D
	50295							
	50296							
	50297							
	50298							
	50299							
	50300							
	50301							
	50302							
	50303							
	50304							
	50305							
	50306							
	50307							
	50308							
	50309							

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EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	DWG REC'D	EQUIPMENT REC'D
Panel Q	50310	✓	✓	✓	✓	✓		
	50311							
	50312							
	50313							
	50314							
	50315							
	50316							
	50317							
	50318							
	50319							
	50320							
	50321							
	50322							
	50323							
	50324							